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**Physical Properties of Some Nickel-containing Corrosion-Resisting Steels**

| General Type of Steel* | | 17Cr-2Ni | 13Cr-13Ni | 18Cr-8Ni | 18Cr-8Ni-Ti | 18Cr-8Ni-Nb | 18Cr-8Ni-1/2Mo | 18Cr-8Ni-2/3Mo | 18Cr-18Ni-2/4Mo-2/3Cu | 15Cr-5·5Ni-1·5Mo-1·5Cu-Nb (or Ti)† |
|---|---|---|---|---|---|---|---|---|---|---|
| **Specifications:** B.S. 970—En | | 57 | 58D | 58A, 58E | 58B | 58F | 58H | 58J | — | — |
| DTD | | — | — | 189 | 189, 171B, 571 | 171B, 571 | — | — | — | — |
| B.S. Aircraft—S | | 80 | — | — | 110, 520, 521 | 110, 520, 521 | — | — | — | — |
| Nearest Equivalent AISI | | 431 | — | 302 | 321 | 347 | — | 316 | — | — |
| **Physical Properties:** | | | | | | | | | | |
| Structure | | Martensitic | Austenitic | Austenitic | Austenitic | Austenitic | Austenitic | Austenitic | Austenitic | Martensitic |
| Specific Gravity | | 7·72 | 8·0 | 7·90 | 7·90 | 7·93 | 7·85 | 7·96 | 8·04 | 7·80 |
| Electrical Resistivity *microhms/cm./sq. cm.* | 20°C. | 72 | 72 | 71 | 72 | 72 | 76 | 75 | 85 | 85 |
| Temperature Coefficient of Electrical Resistance per deg. C. | 20°-100°C. | 0·0011 | 0·0011 | 0·0012 | 0·0012 | 0·0012 | 0·0010 | 0·0011 | — | — |
| Magnetic Permeability | | 210 (max.) | 1·005/1·03 | 1·005/1·03 | 1·03/2·0 | 1·005/1·03 | 1·005/1·03 | 1·05 | 1·005/1·03 | 110 (max.) |
| Melting Point (approx.) | °C. | 1470 | 1450 | 1440 | 1430 | 1430 | 1440 | 1420 | 1375 | — |
| Specific Heat *cals./gm./deg. C.* | 20°-100°C. | 0·12 | 0·12 | 0·12 | 0·12 | 0·12 | 0·12 | 0·12 | 0·12 | — |
| Thermal Conductivity *c.g.s. units* | at 20°C. | 0·045 | 0·035 | 0·036 | 0·037 | 0·037 | 0·038 | 0·039 | 0·031 | 0·038 (50°C.) |
| | at 100°C. | 0·049 | 0·037 | 0·039 | 0·039 | 0·039 | 0·040 | 0·040 | — | — |
| | at 400°C. | 0·060 | 0·045 | 0·049 | 0·049 | 0·049 | 0·049 | 0·049 | — | 0·055(450°C.) |
| Average Coefficient of Thermal Expansion *millionths/deg. C.* | 20°-100°C. | 10·3 | 18·0 | 17·0 | 16·9 | 17·0 | 16·5 | 16·5 | 17·0 | 12·0 |
| | 20°-300°C. | 11·0 | 18·3 | 17·8 | 17·7 | 18·0 | 17·2 | 17·3 | — | 13·0 |
| | 20°-500°C. | 11·7 | 19·0 | 18·4 | 18·3 | 18·6 | 18·0 | 18·0 | — | 13·0 |
| | 20°-800°C. | — | 19·8 | 19·7 | 19·6 | 19·6 | 19·0 | 19·4 | — | — |

**Mechanical Properties of Some Nickel-containing Corrosion-Resisting Steels**

| General Type of Steel * | 17Cr-2Ni | 13Cr-13Ni | 18Cr-8Ni | 18Cr 8Ni-Ti | 18Cr-8Ni-Nb | 18Cr-8Ni-1/2Mo | 18Cr-8Ni-2/3Mo | 18Cr-18Ni-2/4Mo-2/3Cu | 15Cr-5·5Ni-1·5Mo-1·5 Cu-Nb (or Ti) † | |
|---|---|---|---|---|---|---|---|---|---|---|
| Heat-Treated Condition | O.Q. 980°C. T. 650°C. | Fully Softened | Fully Softened | Fully Softened | Fully Softened | Fully Softened | Fully Softened | Fully Softened | Precipitation Hardened ‡ | Hardened and Tempered¶ |
| **Mechanical Properties 20°C.** | | | | | | | | | | |
| 0·5% Proof Stress, *tons/sq. in.* | 45 | 15 | 18 | 18 | 17 | 17 | 18 | 18 | 65 (0·1%) | 54 (0·1%) |
| Maximum Stress, *tons/sq. in.* | 58 | 38 | 42 | 43 | 42 | 42 | 42 | 39 | 76 | 63 |
| Elongation, *per cent* | 23 | 50 | 50 | 50 | 57 | 53 | 50 | 47 | 15 | 25 |
| Reduction of Area, *per cent* | 55 | 50 | 50 | 50 | 62 | 50 | 50 | 66 | 50 | 60 |
| Maximum Shear Stress, *tons/sq. in.* | 36 | 34 | 39 | 36 | 36 | 36 | 37 | — | — | — |
| Fatigue Limit, *tons/sq. in.* (unbroken after $10^7$ reversals) | 24 | 15·5 | 17 | 17·5 | 19·5 | 17·5 | 17·5 | 17 | 38 | 38 |
| Modulus of Elasticity *millions of lb/sq. in.* | 30·5 | 29 | 29 | 29 | 29 | 29·5 | 29·5 | 29 | 29 | — |
| Impact Strength, *ft.-lb.* Izod | 45 | 100 | 100 | 90 | 90 | 90 | 90 | 80 | 25 | 75 |
| Hardness, Brinell | 270 | 165 | 170 | 175 | 175 | 170 | 180 | 160 | 380 | 310 |
| **Machining Properties:** | Good | Fair | Fair | Fair | Fair | Fair | Fair | Fair | Good | |
| **Welding Properties:** | Poor. Welds brittle and little response to heat-treatment | Good, but in the welded condition should only be used under mildly corrosive conditions | Good, but in the welded condition should only be used under mildly corrosive conditions | Good. Heat-treatment after welding not necessary | Good. Heat-treatment after welding not necessary | Good. Heat-treatment after welding not necessary | Good. Heat-treatment after welding not necessary | Good. Heat-treatment after welding not necessary | Good. Where necessary, a heat-treatment of 2 hrs. at 560°C. may be carried out after welding | |
| **Softening Temperature °C.** | 650 Air cool | 950-1050 Cool quickly | 1050 Cool quickly | 1050 Cool quickly | 1050 Cool quickly | 1050 Cool quickly | 1050 Cool quickly | 1050 Cool quickly | | |

N.B.—The above properties relate to materials in bar form. In certain cases 'free-machining' grades are also available.

* The term 18Cr-8Ni is used to denote general types, but in some cases, particularly the molybdenum-containing steels, the nickel content may be appreciably higher than 8%.

† Firth-Vickers F.V. 520(B) Steel.

‡ Normalized 1020°C., air-cooled to room temperature + 2 hrs. 750°C., air-cooled; not less than 2 hrs. at room temperature; 4 hrs. 450°C.

¶ Normalized 1020°C., air-cooled to room temperature + 2 hrs. 750°C., air-cooled; not less than 2 hrs. at room temperature; tempered 2 hrs. 560°C.

# ABSTRACTS OF CURRENT PUBLISHED INFORMATION ON NICKEL AND ITS ALLOYS

## NICKEL

### Effect of Creep Strain on Ductility of Nickel

D. KRAMER and E. S. MACHLIN: 'The Effect of High-Temperature Strain on Crack Formation and Ductility in Commercially Pure Nickel.'

*Trans. Metallurgical Soc., Amer. Inst. Mining and Metallurgical Engineers*, 1959, vol. 215, Feb., pp. 110–12.

The authors briefly review the mechanisms which have been proposed to explain the process of intercrystalline cracking in metals during high-temperature creep. The experimental work reported consisted in pre-stressing of tensile specimens of nickel, at 920°C., for various amounts, at a rate of 1 per cent. per hour, followed by tensile testing at −196°C., at a strain rate of 25 per cent. per hour until rupture. The high-temperature tests were conducted in a hydrogen atmosphere, and for the sub-zero determinations the specimens were surrounded by liquid nitrogen.

Finally, specimens were metallographically examined, in comparison with samples which had been strained at the high temperature only.

The results show that sub-zero ductility decreases linearly with increase in prior high-temperature creep strain. The effect is correlated with formation of intercrystalline cracks and voids during the high-temperature creep strain: the magnitude of the decrease is believed to be a function of the notch-sensitivity of the material.

### Influence of Environment on the Creep Properties of Nickel

P. SHAHINIAN and M. R. ACHTER: 'A Comparison of the Creep-Rupture Properties of Nickel in Air and in Vacuum.'

*Trans. Metallurgical Soc., Amer. Inst. Mining and Metallurgical Engineers*, 1959, vol. 215, Feb., pp. 37–41.

This paper relates to one section of an extensive research on the influence of environment on the creep-resistance of nickel and nickel alloys, carried out at the U.S. Naval Research Laboratory, Washington, D.C. The subject matter of the paper is closely similar to that contained in *U.S. Naval Research Laboratory Report No.* 5036, 1957: see abstract in *Nickel Bulletin*, 1958, vol. 31, No. 6, p. 157. (Other related papers were abstracted *ibid.*, Nos. 2 and 11, pp. 50 and 296.)

### Influence of Curie Temperature on Rate of Oxidation of Nickel

H. UHLIG, J. PICKETT and J. MACNAIRN: 'Initial Oxidation Rate of Nickel and Effect of the Curie Temperature.'

*Acta Metallurgica*, 1959, vol. 7, Feb., pp. 111–17.

Data indicating that initial oxidation rates undergo a discontinuity at the Curie temperature were first presented (for nickel-iron alloys) by TAMMANN and SIEBEL (*Zeitsch. anorg. Chem.*, 1925, vol. 148, p. 297) and later literature has confirmed the occurrence of the anomaly in other materials, including nickel. The present authors report a further study on this metal.

Nickel foil was oxidized for 24-hour periods within the temperature range 307° to 442°C., producing NiO films up to a maximum thickness of 3400 Å. A discontinuity in oxide growth was observed at the Curie temperature (353°C.). Data obtained at 390°, 394° and 405°C., as a function of time, show that a two-stage rate of oxide growth occurs, a higher rate following an initially lower rate. Both stages are represented by a linear relation between oxide thickness and logarithm of time.

The authors offer the following summary with reference to the significance of these data:

'Based on control of the initial oxidation rate by electron transfer from metal to oxide, the discontinuity of oxide thickness at the Curie temperature can be explained by an observed work function of nickel slightly higher above the Curie temperature than below. The change of work function also explains a slightly higher activation energy for oxidation above the Curie temperature than below. Calculations show that the density of trapped electrons in a constant density space charge next to the metal surface is $1{\cdot}3 \times 10^{15}$ at 390°C. Density of available sites for trapped charge in a diffuse charge layer, which grows on top of the constant-density layer, is the same order of magnitude ($5{\cdot}5 \times 10^{15}$). A higher density of sites for NiO than for $Cu_2O$, plus a higher work function for Ni than for Cu, are in line with a lower oxidation rate of Ni compared to Cu. It is calculated that contact of oxide with Ni or Cu lowers the work function at the metal-oxide interface by about 0·6 V. This value agrees, in the case of $Cu_2O$, with the energy required to excite an electron from the filled band to the conduction band.'

### Effect of Hydrostatic Pressure on Magnetization of Nickel

D. GUGAN: 'The Change of Spontaneous Magnetization with Hydrostatic Pressure.'

*Proc. Physical Soc.*, 1958, vol. 72, Dec. 1, pp. 1013–26.

Report of measurements of the effect of pressure on spontaneous magnetization of nickel (rolled, and rolled-and-annealed), cobalt, a nickel-copper alloy containing 11·2 at.% copper, and nickel-iron alloys containing 30, 36, 48 and 50% nickel. The measurements were made by means of a thin disc permeameter designed to produce high magnetic fields for very small power consumption. The results were found to be in good agreement with those reported in other hydrostatic-pressure experiments and with data obtained in investigations of thermodynamically related 'forced' volume magnetostriction. Under some conditions nickel shows an anomalously large, linear, decrease of spontaneous magnetization with pressure. Possible reasons for this effect are discussed.

### Magnetostriction in Nickel at Low Temperature

W. D. CORNER and F. HUTCHINSON: 'The Saturation Magnetostriction of Nickel Crystals at Low Temperatures.'

*Proc. Physical Soc.*, 1958, vol. 72, Dec. 1, pp. 1049–52.

The saturation magnetization of two single crystals of nickel was measured over the temperature range 20° to 500°K. (−253° to 227°C.) by a capacitance-bridge method. Resultant values are compared with those reported by other investigators.

### Ion-Exchange Procedure for Determination of Nickel

L. E. HIBBS and D. H. WILKINS: 'The Determination of Aluminium, Titanium and Nickel in their Alloys.'

*Talanta*, 1959, vol. 2, Jan., pp. 16–20.

In the method described, aluminium, titanium and nickel are absorbed on a cation-exchange column which is coupled with an anion-exchange column. A 0·8*M* hydrofluoric/0·06*M* hydrochloric-acid mixture elutes aluminium from both columns, transfers titanium to the second column and leaves nickel in the first column.

The method also gives an improved ion-exchange method for the separation of mixtures of nickel, aluminium, cobalt, copper and iron, eliminating preliminary separation of copper. Its use is demonstrated as applied to a permanent-magnet alloy of the following composition: iron 35, cobalt 34, nickel 14·5, aluminium 7, copper 4·5, titanium 5, per cent., and notes are added on application of the procedure to nickel-base alloys containing 1–5 per cent. of aluminium and titanium.

A metalfluorechromic indicator is used to facilitate the titration of an excess of E.D.T.A. for the determination of the elements which form highly coloured complexes.

### Determination of Nickel in Uranium

U.K. ATOMIC ENERGY AUTHORITY, INDUSTRIAL GROUP: 'The Absorptiometric Determination of Nickel in Uranium Metal.'

*Report IGO-AM/S-42*; 1958; 6 pp.

The metal is dissolved in nitric acid and, after the solution has been made alkaline with ammonia solution, the nickel is extracted in chloroform as the dimethylglyoxime complex. The nickel is extracted from the chloroform with dilute acid, and finally determined as the oxidized dimethylglyoxime complex, by measuring its absorbancy, using the Spekker absorptiometer fitted with Ilford 602 filters.

The procedure described is suitable for determination of nickel contents of 5–70 p.p.m.

## ELECTRODEPOSITION AND OTHER COATING METHODS

### Electrodeposition of Nickel from Sulphamate Solutions

D. A. FANNER and R. A. F. HAMMOND: 'The Properties of Nickel Electrodeposited from a Sulphamate Bath.'

*Trans. Inst. Metal Finishing*, 1958/59, vol. 36, Pt. II, pp. 32–42.

The effect of nickel plating in lowering the fatigue limit of steel has been known for many years, and it is also general knowledge that tensile internal stress in an electrodeposit is an important contributory cause of the reduction in fatigue strength. This detrimental effect constitutes a serious limitation to the use of electrodeposited coatings on components subjected in service to critical alternating stresses. The use of deposits of low tensile, or even compressive, internal stress would therefore have obvious advantages.

Reports on the properties of nickel deposits made from sulphamate solutions have indicated that such coatings have a very low tensile internal stress, and that the stress can be made compressive by the use of certain addition agents in the solution. The application of such deposits for electroforming and repair work has already been reported. Additional advantages claimed for the sulphamate solution are rapid rate of plating and a greater hardness in the deposits than can be achieved in coatings deposited from the Watts bath.

In view of these practical advantages, a detailed study was undertaken, in the Armament Research and Development Establishment of the Ministry of Supply, to determine the influence of the respective plating variables on the properties of deposits made from sulphamate baths.

The factors investigated were acidity, current density and temperature, as affecting internal stress, hardness, structure and mechanical properties (including the effect on the fatigue strength of the basis steel). The properties of coatings deposited under the various conditions were compared with those of deposits

from a Watts bath, and the investigation also included assessment of the controllability of sulphamate solutions. In order to obtain, for the fatigue and mechanical tests, deposits free from pits, the addition of a wetting agent was required, and the effect of this reagent, on the properties of the deposits, was also studied.

The methods used in preparation of the sulphamate solution are described: its composition (after purification by the method recommended by Diggin) is given as:

| | | *g./L.* |
|---|---|---|
| Nickel sulphamate | $[Ni(NH_2.SO_2.O)_2]$ | 341 |
| Nickel chloride | $[NiCl_2.6\,H_2O]$ | 3·3 |
| Boric acid | $[H_3BO_3]$ | 29·9 |
| Ammonium ion | $[NH_4{}^+]$ | 0·4 |
| Cobalt | $[Co^{++}]$ | 0·06 |
| Copper | $[Cu^{++}]$ | 0·003 |
| Zinc | $[Zn^{++}]$ | Trace (not determined) |

Internal stress in the deposits was measured by the bent-strip method, using spring-steel strips (0·8% carbon), 7 in. (17·5 cm. long), 0·2 in. (5 mm.) wide, 0·015 in. (0·375 mm.) thick. Procedure for measurement of stress was according to the method described *ibid.*, 1955, vol. 32, p. 85. The strips were stress-relieved, before plating, by heat-treatment for one hour at 620°C.

The basis steel used for study of fatigue-resistance was 80-ton nickel-chromium-molybdenum type to B.S. En 25. Fatigue testing was carried out in a normal Wöhler-type rotating-beam machine. Electroformed nickel sheets were prepared by plating onto a filmed copper base, from the sulphamate and the Watts nickel solutions used for preparing the fatigue specimens.

The results of the individual series of experiments are reported in detail. In general, they revealed a close similarity between the effects of the experimental variables on deposits made from the two types of solution. The following points are selected for special mention:

'(1) To ensure reliable anode corrosion it is essential to include chloride in the composition of a nickel-sulphamate bath. This should be minimized to keep the tensile internal stress of the deposit at a low level: a concentration of 3·3 g./L. nickel chloride ($NiCl_2.6H_2O$) was found to be sufficient.

'(2) The claim for low internal stress in nickel deposits from the sulphamate bath, even when no stress-reducing agent is present, is confirmed.

'(3) Optimum conditions for minimum stress in the sulphamate solution investigated were: acidity pH 4·0; temperature 50°C. Current density, within the range 20 - 60 amp./sq. ft. (2·16-6·48 amp./dm²) was not critical.

'(4) Apart from the low internal stress, the properties of nickel deposits from the sulphamate bath, including hardness, ultimate tensile strength, elongation and microstructure, are almost identical with those of deposits from the Watts-type solution produced under similar conditions.

'(5) The effects of the operating variables in the sulphamate bath upon the properties of the deposit conform broadly with the effects observed in a Watts bath. Acidity above a critical pH has a major effect on hardness, structure and internal stress. Current density, within the normal operating range, has relatively little effect. Temperature had negligible effect on the hardness and structure, except at the lowest temperature investigated (30°C.), but, as for the Watts bath, the internal stress was minimum at 50°C.

'(6) The improvement in fatigue strength of steel plated in the sulphamate bath, resulting from the lower internal stress, was marginal in comparison with the fatigue-strength loss resulting from plating in a Watts solution.

'(7) Under equivalent operating conditions, the sulphamate solution, without the addition of a wetting agent, is more liable to produce pitted deposits than a Watts-type solution.

'(8) While the high deposition rate and low stress of sulphamate nickel deposits make this process attractive for certain applications, e.g., electroforming, the marginal advantage in terms of fatigue strength as compared with older established solutions is not sufficient to compensate for the increased cost and liability to pitting of the sulphamate bath operated without wetting agent.'

*See also:*

**Analysis of Nickel-Sulphamate Plating Solutions**

G. H. BUSH and D. G. HIGGS: 'The Analysis of Nickel-Sulphamate Plating Solutions', *ibid.*, pp. 43-7.

This paper describes the methods of analysis employed for control of the plating solutions used in the experimental work described in the previous abstract. Recommended procedure is detailed for determination of total nitrogen, free ammonia, nickel, boric acid and 1 : 3 : 6 trisulphonic acid.

The methods described have been tested on synthetic solutions of known composition: typical results are shown in the paper.

**Measurement of Porosity in Electrodeposited Coatings**

M. CLARKE and S. C. BRITTON: 'An Electro-Chemical Method of Obtaining an Index of Porosity of Metal Coatings.'

*Trans. Inst. Metal Finishing*, 1959, vol. 36, Pt. II, pp. 58-66.

After comment on the inherent limitations of various methods of evaluating porosity in metal coatings, the authors direct attention to the potential usefulness of the technique developed by SHOME and EVANS (*Jnl. Electrodepositors' Tech. Soc.*, 1951, vol. 27, pp. 45-64), which consists essentially in measuring

the corrosion occurring at pores in a coating under conditions such that the rate of corrosion is controlled by the anodic polarization of the basis metal exposed at the pores. Evidence had been provided that the required condition was approached for nickel coatings on steel by connecting the coated metal, immersed in 3 per cent. NaCl solution, to a copper-gauze cathode of sufficient size to remove the corrosion process from cathodic control. In attempts to extend the application of that method to determination of porosity in other coatings, however, difficulties were encountered in realizing the essential condition of anodic control of corrosion. In particular, no suitable auxiliary cathode could be found for tin-nickel coatings, and it appeared likely that, for a range of coatings, it might be easier to achieve conditions in which the current passed by the coated metal as anode was controlled predominantly by the resistance of an electrolyte filling the pore channels.

This paper reports development of such a procedure, describing materials and apparatus used, the range of experimental conditions considered, and check tests made to verify the accuracy of the porosity rating given by the new test. The essential features of the test, and the conclusions reached are summarized below.

When steel carrying porous coatings of tin-nickel, nickel or 'speculum' is made anodic in 3 per cent. NaCl solution so as to displace the potential of the coating by not more than 100 mV, current is passed almost entirely by the steel exposed at pores. This applies also, over a more limited range, to steel coated with copper and to steel coated with tin, in solutions containing 20 g./L. ammonium thiocyanate and 50 ml./L. of 20-vol. hydrogen peroxide. In such conditions displacement of the potential, measured at the surface of the coating, varies linearly with applied current density. The gradient of the line relating applied current to potential shift varies with the conductivity of the electrolyte and is believed to represent the electrical resistance of the electrolyte contained in the channels of the pores.

No new pores are created during the time needed to establish the current/potential relationship and existing pores are not substantially enlarged. Repeated tests on the same panel tend to produce increasing values of apparent resistance, but the addition of 0·1 per cent. of 'Rochelle Salt' to the NaCl test solution is useful in preventing this effect.

In view of these findings, the measurement of the ratio of shift of potential to applied current density, over a limited range, in solutions appropriate to the metal under test, is considered to be a useful index of porosity which, being a resistance value, depends on the number, area, length and shape of the pores.

Application of the proposed method gives satisfactory indications of the decline of porosity with increasing thickness of coating and of the effects, on porosity, of pre-treatment of the basis metal.

The method developed for coatings of tin-nickel alloy on steel can be applied also to such coatings on copper or nickel.

The authors consider that although the method is not suitable as a routine porosity test it is likely to be helpful in research concerned with the effect of processing variables.

**Effect of Nickel Flashing on Adherence of Enamels**

H. CRYSTAL and G. BULLOCK: 'Oxide Adherence and Nickel Flashing in Vitreous Enamelling.'

*Jnl. Amer. Ceramic Soc.*, 1959, vol. 42, Jan. 1, pp. 30–9.

The ultimate aim of the investigation with which this report is associated was development of a method for direct application of a single cover-coat enamel to sheet steel.

Previous work had shown that during firing the presence of iron oxide at the interface of enamel on steel is essential to adherence. Under controlled laboratory conditions it was found possible to obtain good adherence of a directly applied cover-coat enamel to pre-oxidized sheet steel, and the tests showed interdependence of the amount of oxide, the duration of firing and the temperature. (Without pre-oxidation, however, adherence was poor.) Attempts to obtain equal adherence in enamelling on an industrial scale were not successful, due to the impossibility of maintaining the necessary critical control of the processing variables.

The presence of cobalt and nickel oxides in ground coats is well known to promote adherence of enamel, and a survey of work done in this field has led to the conclusion that the oxides in the enamel are reduced by the iron during firing, and therefore cause oxidation, which favours adherence. If the firing times are sufficiently prolonged the cobalt and nickel may be re-oxidized, an effect which has been observed in practice.

It had thus already been established, at the time of initiation of the research described in this paper, that oxidation was essential for adherence, but the mechanism of adhesion had not been fully elucidated. A programme was therefore drawn up to cover study of other contributory factors. Major interest centred on the rôle of nickel, and the paper opens with a survey of the literature concerned with the following relevant aspects of the subject: nickel flashing and adherence of enamels; iron-nickel alloys and their oxidation characteristics; nickel-flashed steel and its oxidation characteristics.

The work was carried out in four phases:

(1) Investigation of the influence, on oxide adherence, of flashing with nickel or iron after pickling, for various times and at various temperatures, in hydrochloric- or nitric-acid solutions.

(2) Determination of the weight of nickel deposited (in 1–15 minutes) after pickling in acid.

(3) Determination of the amount, character and adherence of the oxide film produced on specimens pickled in hydrochloric or nitric acid, nickel-flashed for times varying up to 15 minutes, and heated at 850°C. for 20 .40, 60, 80, 100 or 200 seconds.

(4) Determination, by impact testing, of the adherence of a cover-coat enamel applied to specimens subjected to the same pre-treatments as in (3), and fired for 4 minutes at 850°C.

The results of the tests are presented *in extenso*, with many illustrations.

The authors' main findings not only confirm the necessity of ensuring that an optimum degree of oxidation occurs, but also establish a relationship between the adherence of the oxide and that of the enamel. The results demonstrate that nickel flashing tends to promote adherence of oxides to the steel sheet, and also promotes adherence of the enamel. Under laboratory conditions it was found possible, when using a nickel-flash treatment, to achieve adherence of a commercially acceptable standard for enamels directly applied, but the surface texture of the enamel was not entirely satisfactory. A relationship was observed between the iron content of a hydrochloric-acid pickling bath and the weight of nickel subsequently deposited during flashing. The suggestion is made that hydrogen absorbed during pickling is also concerned in the nickel-flashing operation

It is considered possible that better control of pickling and nickel-flashing treatments would, by ensuring more uniform deposition of nickel, make possible direct application of a cover-coat enamel in commercial operation.

### 'Composite Electrochemical Materials': Nickel/Silicon-Carbide Electrodeposits

A. E. GRAZEN: 'Plating Process Co-Deposits Oxides or Carbides.'

*Iron Age*, 1959, vol. 183, Jan. 29, pp. 94–6.

The article gives further particulars of the properties obtainable in the thin hard composite coatings (comprising finely divided particles of hard carbides or oxides in a metallic matrix) electrodeposited by the 'CEM' process referred to in *Nickel Bulletin*, 1959, vol. 32, No. 1, p. 10.

Much of the discussion relates to silicon-carbide/nickel coatings. Photomicrographs illustrate the structures of aluminium-oxide/nickel, tungsten-carbide/nickel, silicon-carbide/nickel and silicon-carbide/silver coatings.

## NON-FERROUS ALLOYS

### The Nickel-Titanium-Carbon System

E. R. STOVER and J. WULFF: 'The Nickel-Titanium-Carbon System.'

*Trans. Metallurgical Soc., Amer. Inst. Mining and Metallurgical Engineers*, 1959, vol. 215, Feb., pp. 127–36.

The importance of titanium carbide as a component of cermets is now widely recognized in relation to high-temperature engineering, and the nature of TiC/Ni or TiC/Co composites is being widely studied. Recent investigations have emphasized the importance of control of the grain-size and distribution of the carbide constituent, and of ensuring a thermal-expansion match between the phases involved.

The aim of the authors' study was determination of the phase compositions occurring during the sintering or infiltration process and during subsequent heating of composite materials of these types. Study was confined to the pure ternary system nickel-titanium-carbon, with main interest on the nickel corner of the diagram, since the nickel solid solution is the continuous phase in compositions which show the highest strengths. The paper is introduced by a review of the most recent work on the three relevant binary systems.

The experimental work reported, which is copiously illustrated by diagrams and photomicrographs, indicates that no ternary compounds exist in the Ni-Ti-C system above 870°C.: each of the Ni-Ti intermetallic phases is in equilibrium with TiC. Solution of carbon in $Ti_2Ni$ is only a few atomic per cent., but is sufficient to raise the solidus temperature, enlarge the lattice parameter and increase the microhardness. Solubility of carbon in both TiNi and $TiNi_3$ is very low. At 1307°C. a quasi-binary eutectic occurs between TiC and a nickel solid solution rich in titanium. Ternary eutectics occur on either side, with $TiNi_3$ at 1295°C. and with graphite at 1270°C. The nickel-graphite eutectic is close to 1328°C.

Solubility of TiC + graphite in nickel varies as follows (in atomic per cent.):

3·4 Ti + 2·6 C at 1260°C.
3·1 Ti + 1·2 C at 1000°C.

to about

2·0 Ti + 0·2 C at 600°C.

Thus, if graphite is to be avoided during sintering, the binder of a TiC-Ni cermet must contain an excess of titanium or of some other carbide-forming element.

### Precipitation Phenomena in Nickel-base Alloys

See abstract on p. 115.

### Effect of Hydrostatic Pressure on Magnetization of Nickel-Copper Alloys

See abstract on p. 107.

### Heat Capacity of Low-Chromium Nickel-Chromium Alloys

R. HULTGREN and C. LAN: 'The Heat Capacity of Dilute Solutions of Chromium in Nickel.'

*Trans. Metallurgical Soc., Amer. Inst. Mining and Metallurgical Engineers*, 1959, vol. 215, Feb., pp. 165-6.

Report of determinations on four binary alloys containing, respectively, 1·82, 2·47, 4·38 and 11·03 at. per cent. of chromium: these data are supplemented by values for nickel and for chromium. The results of the measurements show that, apart from ferromagnetic effects, Kopp's law of additivity of heat capacities is approximately followed. The authors' findings show some disagreement with those reported by FOSTER (*Philosophical Mag.*, 1934, vol. 18, p. 470).

### 'Monel' Roofing

'Nickel-Copper Alloy Roof Protects Museum's Treasures.'

*Reprint* from *American Artisan*, 1958; 6 pp.
Issued by INTERNATIONAL NICKEL CO., INC.

The Metropolitan Museum of Art in New York City houses one of the most extensive and varied art collections in the world: the total number of items is estimated at 100,000. As in other museum buildings, roofing material is an important consideration, particularly in the interests of preventing leakage.

In 1936, when some repair became necessary, portions of the roof were replaced by 'Monel' sheet, and the experiment proved so successful that the same material has subsequently been used as further repair was required. Plans are now in hand for completion of re-roofing. The alloy is used in the form of crimped sheeting, thus obviating the need for expansion joints. Some 112 tons of 'Monel' have already been used on the scheme, including installation of flat and standing roofing; base, coping and cap flashings, gutters; skylight trim and frames; downspouts, hatchways, fastenings, ventilators, louvres, etc.

The mechanical strength of 'Monel' and its ductility, combined with high resistance to atmospheric corrosion, ensure trouble-free service, and aesthetically the effect is pleasing because the original soft dull matte finish of the material weathers to a neutral colour which tones well with the slate walls of the building. A further advantage, from the architectural point of view, is that the thermal coefficient of expansion of 'Monel' matches very nearly with that of both steel and concrete, and the alloy has been successfully used for both new roofing and replacement, in conjunction with tile, slate and composition materials. It may also be joined with copper or lead-coated copper, without risk of galvanic corrosion.

This article makes some practical recommendations with regard to installation of 'Monel' roofing and includes illustrations of typical uses.

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## NICKEL-IRON ALLOYS

### Effect of Hydrostatic Pressure on Magnetization of Nickel-Iron Alloys

See abstract on p. 107

### Oxidation of Nickel-Iron Alloys

S. W. KENNEDY, L. D. CALVERT and M. COHEN: 'Oxidation of Three Iron-Nickel Alloys and Iron at 800°C.'

*Trans. Metallurgical Soc., Amer. Inst. Mining and Metallurgical Engineers*, 1959, vol. 215, Feb., pp. 64–72.

It is pointed out that although film formation has been previously investigated by high-temperature electron-diffraction techniques, no record exists of the study of scaling by methods permitting examination of the scale during formation at the reaction temperature. Techniques involving quenching of scales are based on the working assumption that no significant changes take place on cooling: it is believed, however, that during cooling the compounds may change in proportion and that the different thermal expansions of the respective constituents may cause disruption of the scale; it is known that some scales cannot be quenched without spalling. These difficulties are avoided by X-ray-diffraction examination at the temperature of scale formation, and such procedure also provides a method of determining the kinetics of formation of each phase separately, in a multi-component scale.

In the research now reported a study was made of the scaling of three binary iron alloys containing, respectively, 25·6, 75 and 84 per cent. by weight of nickel. Exposure was at 800°C. in oxygen, at atmospheric pressure. A Norelco Geiger-counter diffractometer was used, and, in addition, normal metallographic examination and chemical and X-ray analyses were made, and weight-gain/time curves were obtained. The oxidation of iron was similarly studied. (For a description of the furnace used in the experiments, reference is directed to the *Journal of Scientific Instruments*, 1958, vol. 35, p. 529.)

Results of the various methods of investigation are in essential agreement with regard to orientation, and the order and identity of the layers of scale. There was definite evidence for orientation of the $Fe_2O_3$ layer on iron and on the alloys containing 25 and 75 per cent. of nickel, and this orientation will affect the rate of oxidation when diffusion through the $Fe_2O_3$ layer is a controlling factor.

Use of the high-temperature X-ray furnace made possible identification of FeO and it was also noted that under the conditions of cooling used in these experiments free iron, from decomposition of FeO, was detectable at 475°C.

On the 25 per cent. nickel alloy a small amount of NiO was found, and larger quantities (of the order of 50 per cent. of the scale thickness) were found on the 75 and 84 per cent. nickel alloys. The spinel layer of all the alloys contained nickel ion as one of its constituents. The thickness of the $Fe_2O_3$ decreased with increasing nickel content and was only barely detectable on the 84 per cent. alloy.

Deep sub-scale formation was observed only on the 25 per cent. nickel alloy. This appeared to be associated with cracking of the layers of scale and subsequent penetration of oxygen along the grain boundaries. On the higher-nickel alloys the amounts of sub-scale formation and intergranular oxidation were smaller.

The report is well illustrated with photomicrographs showing scaling and internal-oxidation effects.

### Ion-Exchange Procedure for Analysis of Magnet Alloys

See abstract on p. 107.

## CAST IRON

### 'Ni-Resist': Properties and Applications

INTERNATIONAL NICKEL CO., INC.: 'Engineering Properties and Applications of Ni-Resist.'
5th Edn., Published 1958; 68 pp.

The first section of this publication contains a comprehensive compilation of reference information on the composition and properties of seven grades of high-alloy cast irons containing nickel within the range 13·5 to 36 per cent., with or without copper and/or chromium.

The second section reviews (with many illustrations) typical applications of the 'Ni-Resist' irons in chemical, engineering and other industries. The information on properties and uses is supplemented by tabular corrosion data based on service and laboratory tests of 'Ni-Resist' (and other materials) in some 500 corrosive environments.

### Oxidation-Resistance of Cast Irons

J. A. CAMERON: 'Oxidation of Plain, Alloyed and Ductile Irons at 1300°F.'
*Reprint* from *Corrosion*, 1954, vol. 10, pp. 295–8.
Issued by INTERNATIONAL NICKEL CO., INC., 1958; 4 pp.

Oxidation tests in air at 1300°F. (704°C.), continued for periods up to 4,000 hours, were made on flake-graphite grey cast iron, S.G. iron of normal composition, and highly alloyed cast irons of both flake- and spheroidal-graphite types. The results demonstrate the superior oxidation-resistance of the S.G. irons. (For a full abstract of this paper see *Nickel Bulletin*, 1954, vol. 27, No. 11, p. 210.) The reprint includes discussion by K. D. MILLIS, also referred to in the original abstract.

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## CONSTRUCTIONAL STEELS

### Influence of Microstructure on Fatigue Properties of Nickel-Chromium-Molybdenum High-Strength Steels

H. E. FRANKEL, J. A. BENNETT and W. A. PENNINGTON: 'Fatigue Properties of High-Strength Steels.'
*Trans. Amer. Soc. Metals*, 1958, vol. 52, *Preprint* 121, 23 pp.+ tables and figs.

The work reported was initiated in an attempt to resolve some of the conflicting conclusions drawn by various investigators with respect to the influence, on the fatigue characteristics of steels, of metallurgical variables: in particular, the presence of retained austenite in tempered martensite.

A study was made of variation in fatigue properties, as a function of carbon content (0·44–1·06 per cent.), heat-treatment, hardness, retained austenite, and refrigeration treatment. The steels used were two of low-alloy nickel-chromium-molybdenum type and two low-alloy tool steels of the manganese-molybdenum and chromium types, respectively. Fatigue specimens were prepared by a technique which, involving machining, grinding, polishing, copper plating, heat-treating and final removal of the copper deposit, was intended to minimize possible masking effects of the usual grinding-to-size after heat-treatment.

All the specimens were austenitized for one hour at 1550°F. (842°C.) and then subjected to one of six heat-treatments: direct quenching and tempering; 'Marquenching' and tempering; direct quenching, refrigeration and tempering; 'Marquenching', refrigeration and tempering; direct quenching, refrigeration and double tempering; 'Marquenching', refrigeration and double tempering. The resultant microstructures showed, in every case, tempered martensite, varying amounts of retained austenite, and carbides. Specimens were fatigue tested to fracture. In a supplementary study, the effect of fatigue testing on retained austenite was investigated.

The conclusions drawn from the results obtained are summarized as follows:

'Moderate amounts of alloying elements, except carbon, are not so important as microstructure, in their effect on fatigue strength.

'Fatigue strength is a linear function of hardness up to some limiting value, which depends on the carbon content of the steel. Beyond that point there appears to be a very rapid decrease in fatigue strength.

'Refrigeration prior to tempering was found to improve fatigue properties.

'Retained austenite up to about 10 per cent. lowered fatigue strength, but beyond that percentage there was apparently little further effect.

'Fatigue stressing transforms retained austenite to an untempered martensite; the amount so formed is an indication of the stress applied. At equal stress levels there is a decrease in the amount of transformation as the carbon content of the steel is increased.

'The hardness required to attain a given fatigue strength is greater as the carbon content of the steel is raised.

''Marquenching' was not found to confer any benefit additional to that obtained by direct quenching.'

The reason for the deleterious effects of retained austenite is discussed and the following hypothesis is advanced: high localized stresses resulting from an austenite→martensite reaction during fatigue may nucleate sub-microscopic cracks which lower the fatigue-resistance of the steel. Transformation of retained austenite before cyclic stressing was found to increase fatigue-resistance.

### 9% Nickel Steel for Low-Temperature Service

T. N. ARMSTRONG, J. H. GROSS and R. E. BRIEN: 'Properties Affecting Suitability of 9 Per Cent. Nickel Steel for Low-Temperature Service.'
*Welding Jnl.*, 1959, vol. 38, Feb., pp. 57s–65s; disc. by (1) W. L. NEELY, and (2) C. E. HARTBOWER and W. P. HATCH, pp. 65s–70s; authors' reply, p. 70s.

This is the final form of the paper abstracted,

in preprint form, in *Nickel Bulletin*, 1958, vol. 31, No. 6, pp. 166–7.

NEELY reported that welding tests conducted by Lukens on 1-in. plate of 9 per cent. nickel steel agree closely with the results reported by Armstrong *et al.* Preparation and welding technique are described and illustrated. The results obtained by Lukens indicate that the 9 per cent. nickel steel plate can be satisfactorily welded, by the use of either special 'Inconel' electrodes or electrodes of 25–20 chromium-nickel steel. The former type gave the better degree of ductility in the weld metal.

Particulars are given of three butt-weld joint designs which have been successfully used, and typical test data are recorded.

HARTBOWER and HATCH reported results of tests in which the Watertown Arsenal low-energy-blow + full-capacity-blow test (*ibid.*, 1957, vol. 36, p. 494s) was applied to the 9 per cent. nickel steel, in three conditions. The results are graphically recorded and their significance is discussed in relation to specification requirements for steels to be used in sub-zero service.

### Influence of Nickel on High-Speed Tool Steels

R. F. HEHEMANN and A. R. TROIANO: 'How Nickel Affects High-Speed Tool Steels.'

*Iron Age*, 1958, vol. 182, Dec. 18, pp. 94–6; Dec. 25, pp. 52–6.

Although the presence of nickel is known to contribute to various properties desirable in constructional steels, it is rarely present as an integral constituent in high-speed tool steels. The reason for this is two-fold: (1) the relatively simple annealing cycles used for standard high-speed steels are not effective in the presence of an appreciable percentage of nickel, and the steels are therefore not readily softened for machining, and (2) nickel-containing steels have a tendency to retention of austenite after quenching, and excessive amounts of retained austenite could inhibit attainment of the degree of hardness essential in tool steels.

It was, however, believed that if composition and treatments could be suitably adjusted, the addition of nickel to this type of steel might confer the benefits which result from its presence in constructional steels; for example, improved ductility and resistance to shock. Such modification might provide a class of high-speed steel not at present available.

Based on this assumption, research was carried out, at the Case Institute of Technology, Cleveland, on two tool steels modified by additions of 3 per cent. nickel. The typical steels selected for the tests were, respectively, of the following classes: 18–4–1 tungsten-chromium-vanadium, and 6–5–4–2 tungsten-molybdenum-chromium-vanadium.

Part 1 of the report describes tests made to determine the influence of the nickel additions on the transformations occurring during annealing of the steels. The experimental work reported resulted in development of annealing procedures suitable for treatment of the two modified steels.

Part 2 is concerned with hardening treatments, which included sub-zero exposure, and with the response of the hardened steels to tempering, using both single and cyclic treatments.

The indications from the work are that by suitable modification in heat-treatment it is possible to obtain, in nickel-containing high-speed steels of the types used, a combination of properties not found in nickel-free steels of comparable compositions.

### Welding Stainless Steel to Carbon and Low-Alloy Steels

J. J. B. RUTHERFORD: 'Welding Stainless Steel to Carbon or Low-Alloy Steel.'

*Welding Jnl.*, 1959, vol. 38, Jan., pp. 19s–25s.

This paper is one of the 'interpretive reports' issued under the aegis of the Welding Research Council. It deals specifically with joining of austenitic steels (mainly the 18–8–Nb type) to constructional carbon and low-alloy types, as required, for example, in components of steam plant. The author traces the history of the practice of such dissimilar-metal welding from the introduction (about 1925) of higher temperatures in steam-generating plant, with resultant need for use of the austenitic steels for superheater tubes and other components working under the more severe temperature conditions. This change, combined with the need, on economic grounds, for retaining the less-highly-alloyed steels for associated portions of the plant, gave rise to the need for joining the dissimilar metals involved.

This report makes a review of the successes which have been achieved in this connexion and of the problems which have arisen. The survey is followed by consideration of some of the research which has been carried out to overcome these problems, and of some service experience in use of the methods which have been proposed for joining the dissimilar metals. Reference is given to original reports from which more detailed information can be obtained on various aspects of the subject.

An appendix to the report contains a summary of recommended practice for welding austenitic to ferritic steels.

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## HEAT- AND CORROSION-RESISTING MATERIALS

### Structural Changes in Nickel-Chromium-Iron Alloy during Creep-Rupture Test

R. J. RAUDEBAUGH and E. P. SADOWSKI: 'Microstructural Changes in a 42% Ni, 30% Cr, 26% Fe Alloy during Creep-Rupture Testing.'

*Trans. Metallurgical Soc., Amer. Inst. Mining and Metallurgical Engineers*, 1959, vol. 215, Feb., pp. 23–9.

The work reported in this paper was undertaken to explain two anomalies which had been observed

in high-temperature tests on an alloy of the composition stated. It had been found that:

(1) for time periods greater than 550 hours this alloy showed better stress-rupture and creep strength at 1800°F. (982°C.) than at 1600°F. (870°C.);

(2) although non-magnetic when placed on stress-rupture test, some specimens had been found, after test, to be magnetic, and it was observed that the gain in magnetic response was not solely a function of time and temperature of testing.

The tests were made on material from a production heat, processed according to commercial practice: the composition is given as carbon 0·05, iron 25·83, chromium 30·13, nickel 41·59, titanium 0·41, per cent. The material was tested in two conditions:

(*a*) Mill-annealed (heated at 1900°F. (1038°C.)) in a continuous furnace for about 30 minutes and air-cooled: grain-size A.S.T.M. 8 or finer.

(*b*) Mill-annealed and subsequently soaked for 2 hours at 2050°F. (1120°C.) in a laboratory furnace and air-cooled: grain-size A.S.T.M. 4-5.

Rupture tests were carried out at 1400°, 1600° and 1800°F. (750°, 870° and 982°C.), in accordance with A.S.T.M. procedures. In tests in which only rupture life and elongation at fracture were determined, specimens of 0·25-in. (6-mm.) diameter and 1-in. (25-mm.) gauge length were used. In tests made to obtain creep curves the specimens were of 0·375-in. (9-mm.) gauge length. Some of the tests under relatively low stress were discontinued short of rupture.

Structural changes which had occurred during testing were determined by examination of the specimens by light microscopy and X-ray diffraction. Diffraction-pattern recordings were made with a diffractometer using copper radiation, on samples heavily etched in a picric-hydrochloric acid mixture, and also on electrolytically extracted residues. Of the various electrolytes used in extraction, best results were obtained with an aqueous solution of NaCl. Nitrogen determinations, on specimens after test, were made at positions adjacent to fracture or, in cases in which the tests were discontinued prior to fracture, on material from the mid-gauge length.

The detailed and well-illustrated report presented leads to the following conclusions:

'(1) Alpha prime, the non-magnetic, high-chromium body-centred-cubic ferrite, formed after very short times at 1400° and 1600°F. (750° and 870°C.). It did not form at 1800°F. (982°C.). The phase first appeared in acicular form within the grain and as blocky, sharply angular particles at grain boundaries and at grain-boundary junctions. With increasing time at test temperature the acicular form gradually went into solution and a simultaneous growth of the sharply angular form of $\alpha$ prime was noted. The decreasing slope of the log stress-log rupture time plot at 1600°F. (870°C.) is believed to be due to the re-solution of the acicular form of $\alpha$ prime, which homogenized the matrix relative to chromium. Grain growth was very slight at 1600°F. (870°C.) but may have made some contribution to decreasing the stress-rupture-life plot.

'(2) The greater rupture strength at 1800°F. (982°C.) than at 1600°F. (870°C.) after about 550 hours is thought to be attributable in part to the relatively rapid grain coarsening encountered at 1800°F. (982°C.) and also to the fact that at this temperature the alloy consists of a single phase, $\gamma$, as opposed to the finer-grained two-phase structure, $\gamma + \alpha$ prime, at 1600°F. (870°C.).

'(3) One constituent present in all conditions of the alloy remained unidentified. It is not believed that it plays any part in the reversal in strength of the alloy at 1600° and 1800°F. (870° and 982°C.).

'(4) The amount of nitrogen pick-up from the atmosphere during testing is related not only to the time and temperature of exposure but also to the number of entry sites for the nitrogen. The source of nitrogen entry may be cracks or fissures formed in the progress of the test, or possibly more grain-boundary entries, such as in a fine-grained as opposed to a coarser-grained condition.

'(5) Magnetic change (from non-magnetic to magnetic) is related to the amount of $Cr_2N$ formed. It is believed that the magnetic change is brought about by depletion of chromium from the matrix, to form $Cr_2N$, leaving a nickel-rich magnetic solid solution.'

**Effect of Creep Strain on Ductility of Nickel**

See abstract on p. 106.

**Creep-Rupture Strength of Chromium-Nickel-Molybdenum Steels in Sheet and Bar Form**

G. V. SMITH, F. GAROFALO, R. W. WHITMORE and R. R. BURT: 'Creep-Rupture Strength of Austenitic Cr-Ni-Mo Steels in Sheet and Bar Form.'

*Amer. Soc. Mechanical Engineers, Paper No.* 58-*A*-102, Dec. 1958; 7 pp.

Appreciable scatter in 100- and 1000-hour creep-rupture tests has been observed in molybdenum-containing 18-8 chromium-nickel steel to A.I.S.I. Type 316 specification. The authors refer to literature bearing on the possible influence of melting practice, composition and thermal and mechanical treatment, and report original experiments in which comparison was made of the creep-rupture strength of 7 heats of bar stock and 7 heats of sheet stock of this type of steel, tested at 1100°, 1300° and 1500°F. (593°, 704° and 815°C.). Full analyses of the individual steels are recorded.

The results of the tests emphasize the significance of the combined carbon+nitrogen+phosphorus content (within the specification limits). Of the

variability found in testing the fourteen steels at the three temperatures, at least 33 per cent., and possibly up to 54 per cent., could be accounted for by variation in the chemical factor C+2·75 N+1·5 P. The additional variability may, it is believed, be associated with differences in thermal and mechanical history and with inherent variability in creep-rupture testing. At all three test temperatures the bar-stock steels were found to be slightly stronger for rupture in 100 hours, and at 1300° and 1500°F. (704° and 815°C.) they were superior also with regard to 1000-hour rupture. At 1100°F. (593°C.) there was no essential difference between the 1000-hour rupture strengths of the bar and sheet steels.

### Influence of Ceramic Coatings on Creep Properties of Nickel-Chromium Alloys

J. R. CUTHILL, J. C. RICHMOND and N. J. TIGHE: 'Effect of a Ceramic Coating on the Creep Behaviour of Some High-Temperature Alloys.'

*Amer. Ceramic Soc. Bull.*, 1959, vol. 38, Jan. 15, pp. 4–12.

The paper presents results of load-extension and creep tests on uncoated and ceramic-coated sheet-metal specimens of 25-20 chromium-nickel stainless steel and of two 80-20-type nickel-chromium alloys. The information is closely cognate with that given by CUTHILL and HARRISON in *Wright Air Development Center, Report* 56-85 (see *Nickel Bulletin*, 1957, vol. 30, No. 7-8, p. 132) which, however, also included the results of similar tests on ceramic-coated nickel specimens.

### Precipitation Phenomena in Nickel-base Alloys

J. MANENC: 'Study of Precipitation in Some Nickel-base Alloys.'

*Acta Metallurgica*, 1959, vol. 7, Feb., pp. 124–30.

Earlier work by the author on nickel-chromium-base alloys containing aluminium and titanium had demonstrated three stages of structural modification involved in the precipitation-hardening reaction: (1) the pre-precipitation condition; (2) an intermediate stage in which the matrix and the precipitate are still coherent, and (3) the stage in which the respective structures are in equilibrium. The present paper reports subsequent investigation of precipitation phenomena in other high-purity nickel-base alloys, of the following types:

| Al % | Ti % | Cu % | Si % | Mo % | Ni % |
|---|---|---|---|---|---|
| 7·8 | — | — | — | — | Balance |
| 5 | 2 | — | — | — | |
| 2 | 7 | — | — | — | |
| 6 | — | 20 | — | — | |
| | | | 7 | — | |
| | | 30 | 5 | — | |
| | | | 5 | 7·5 | |

The observations made show that in all these alloys a face-centred-cubic phase is precipitated in a face-centred-cubic matrix. In all cases the change was a three-stage modification, as in the Ni-Cr-Al-Ti alloys previously examined. The materials examined differ among themselves, in that in some of the alloys the precipitate appears in the form of platelets, whereas in others the particles are cubic. The latter form of precipitate is characteristic of alloys in which the difference between the parameter of the matrix and of the precipitate is of the order of 0·01 Å.

### The Nickel-Titanium-Carbon System

See abstract on p. 110.

### 'D-979' Iron-Nickel-Chromium High-Temperature Alloy

E. E. REYNOLDS and R. K. PITTER: 'Two New Superalloys Excel at Elevated Temperatures.'

*Iron Age*, 1959, vol. 183, Jan. 22, pp. 70–1.

The article gives brief details of two new precipitation-hardenable materials developed by Allegheny Ludlum Steel Corporation. One of them, 'D-979', is produced by consumable-electrode, vacuum-arc melting: the other, 'AF-71', is made by conventional arc-melting methods. The compositions of the new materials are given on p. 116.

Heat-treatment of 'D-979' consists in solution-annealing in the range 1850°–2050°F. (1010°–1120°C.) and double ageing, comprising heating at 1550°F. (840°C.), for 6 hours, followed by treatment at 1300°F. (705°C.) for 16 hours.

Typical room-temperature and high-temperature properties are quoted for both alloys.

### Testing under Combined Static and Dynamic Stress

P. E. HAWKES and C. H. EK: 'A Machine for Evaluation of High-Temperature Alloys under Combined Static and Dynamic Stresses.'

*A.S.T.M. Bull.*, 1959, Jan., pp. 46–50; disc., p. 51.

In operation of a gas-turbine engine the blading is subjected to static tensile stress due to centrifugal forces acting on it, and to flexural fatigue stresses resulting from resonant vibration of the blades. To test materials for such service, and for other applications involving combined creep and fatigue conditions, there is therefore need for a testing machine capable of evaluating, at elevated temperatures, the resistance of small specimens to the simultaneous imposition of creep loading and flexural fatigue stresses.

This paper describes a machine developed, by sections of General Motors Corporation, to meet these requirements. Design and mode of operation are described and well illustrated. The machine is capable of giving steady stresses up to 85,000 p.s.i. (38 t.s.i.; 60 kg./mm.²). together with cyclic flexural stresses up to 45,000 p.s.i. (20 t.s.i.; 31·5 kg./mm.²). Frequency is set sufficiently high to provide for the relatively large number of cycles often experienced

**Compositions of 'D-979' and 'AF-71' Alloys**

(*See abstract on p. 115*)

| Alloy | C % | Si % | Mn % | Cr % | Ni % | Fe % | Mo % | W % | Ti % | Al % | Ni % |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 'D-979' | 0·05 | 0·30 | 0·30 | 15·0 | 15·0 | bal. | 4·0 | 4·0 | 3·0 | 1·0 | — |
| 'AF-71' | 0·25 | 0·30 | 18·0 | 12·5 | — | bal. | 3·0 | — | — | — | 0·20 |

by blades, panels and other light-weight engine components in a given period of exposure. The application of the test is demonstrated by results on two representative high-temperature materials: 'A-286' (complex precipitation-hardening austenitic steel containing aluminium and titanium), tested at 1100°F. (595°C.), and 'G.M.R.-235D' (a nickel-chromium-base precipitation-hardening cast alloy containing aluminium, titanium and molybdenum), tested at 1800°F. (980°C.).

### Heat-Treatment of 'Nimonic' Alloys

W. BETTERIDGE: 'Treatment of 'Nimonic' Alloys for Special Applications.'

*Metal Treatment*, 1959, vol. 26, Feb., pp. 45-52.

The success of the alloys of the 'Nimonic' series in aircraft gas turbines has led to widespread interest in their suitability for components of industrial gas-turbine units. The service conditions in the two fields vary widely, however; for the industrial engines temperatures are lower than those to which the aero engines are subject, and the life required is much longer, e.g., of the order of at least 20,000 hours. Design data and general information on properties must therefore cover a relatively wide range of temperature, and it was to be anticipated that the cycles of thermal treatment which yielded a combination of properties optimum for service at temperatures in the higher range of temperature would not necessarily be best suited for treatment of material to be used in components employed in the lower-temperature conditions.

Some concern has been expressed at the relatively low total creep extension at which alloys of this class fracture under constant-stress, constant-temperature creep conditions, at temperatures in the range 600°-700°C. Tests reported in this paper, however, demonstrate that this relatively low stress-rupture ductility is not associated with either tensile embrittlement or notch sensitivity.

The second part of the paper deals with specialized heat-treatments which have been devised to provide the higher creep extension at fracture called for in certain applications. It is shown that the increased elongation can be obtained by the inclusion of an over-ageing treatment in the range 850°-900°C., depending on the particular alloy. For service at temperatures in the region of 500°C., however, appreciably improved strength can be obtained by an appropriate combination of lattice distortion and precipitation hardening. The lattice distortion can be introduced either by hot-working, if it is found possible to control that procedure with sufficient precision, or by cold-working of the previously solution-treated material.

### Irradiation Tests on Nickel- and Cobalt-base Alloy Springs

R. L. MEHAN: 'Irradiation of 'Haynes-25' and 'Inconel-X' Compression Springs in High-Temperature High-Pressure Water.'

*Amer. Soc. Mechanical Engineers, Paper* 58-*A*-94, Dec. 1958; 7 pp.

Experimental investigations already reported have shown that exposure to neutron bombardment tends, in general, to increase the mechanical strength of metallic materials, and to lower ductility, and it is indicated that these effects are intensified as exposure is prolonged. Under conditions involving environments such as high-temperature water the possible effect of corrosion must also be considered.

The specific conditions studied in the experiments reported in this paper comprised exposure to relatively low neutron fluxes, in high-pressure water at 580°F. (305°C.).

Springs of the nickel-base alloy 'Inconel' and of the cobalt-nickel-chromium-tungsten alloy 'Haynes No. 25' were wound in the cold-drawn condition, heat-treated at temperatures experimentally determined as optimum, and pickled, and were exposed together with cold-straightened, heat-treated and pickled sections of wires of the two alloys.

Irradiation test assemblies and conditions are described and illustrated. Four types of test, and metallographic examination, were carried out before and after irradiation, for 2000 hours, in conditions more intense than those to which the springs would be subject in service.

The post-irradiation tests revealed no serious harmful effects caused by the exposure. For both materials the spring constant had increased: for the 'Haynes 25' alloy the maximum change was 8 per cent., for the 'Inconel' 5 per cent. This alteration is tentatively attributed to changes in the elastic constants. The free length decreased by 2-3 per cent. No cracks or defects were found on the surface of the specimens. The effect of irradiation on the plastic-flow curve was slight, as revealed by three-point loaded bend tests on 0·160-in. (4-mm.) diameter rods irradiated with the springs.

### Cobalt-Nickel-base High-Damping Turbine-Blading Alloys

A. COCHARDT: 'Development of a Ferromagnetic Cobalt-base High-Temperature Alloy.'

*Trans. Amer. Soc. Metals*, vol. 52, *Preprint* 119; 15 pp. + tables and figures.

The damping characteristics of an alloy or a steel govern to a great extent its suitability for use, at various temperatures, as blading material in industrial gas turbines, compressors, and, in particular, in steam turbines operating at partial admissions. High damping capacity reduces susceptibility to fatigue under vibration, and permits the use of higher operational temperatures. The author's previous investigations in the same field had revealed the extremely high internal-damping characteristics exhibited, as a result of magneto-mechanical hysteresis, by certain ferromagnetic alloys of cobalt-nickel base (see abstract in *Nickel Bulletin*, 1957, vol. 30, No. 1-2, p. 6). Such alloys offered promise of development into materials capable of replacing 12 per cent. chromium steel in units designed to operate at temperatures at which the strength of that steel is no longer adequate (i.e., above about 1050°F. (565°C.)). This report gives details of experiments carried out to develop 'a cobalt-base alloy which would have the strength of a superalloy, but which would be ferromagnetic at the operating temperature and would have a high internal damping.'

The following tests were carried out on alloys rendered age-hardenable with titanium and aluminium, and containing from 62 to 88 per cent. of cobalt, varying amounts of nickel, and small percentages of other elements:

*Damping tests* in a torsion pendulum apparatus.

*Creep-rupture tests* at 1200°F. (650°C.).

*Fatigue tests.*

*Cyclic and Isothermal tests*, to determine oxidation-resistance. Cyclic tests involved alternating exposure at 1900°F. (1038°C.) for 7 minutes and air-cooling for 7 minutes; in the isothermal tests the specimens were held at 1200°F. (650°C.) and weighed at weekly intervals.

*Hardness tests* on specimens age-hardened at 1200°, 1300° and 1350°F. (650°, 705° and 732°C.) for periods ranging from 1 hour to 512 hours.

Solution-treatment was carried out, in a hydrogen atmosphere, for 1 hour at either 1900° or 1950°F. (1040° or 1065°C.). Test specimens were usually aged for 24 hours at 1200°F. (650°C.) prior to testing.

The data presented show that the strength and damping characteristics of some of the alloys tested were similar at 1200°F. (650°C.) to those exhibited at 900°-1000°F. (480°-540°C.) by the high-chromium steel conventionally used for steam-turbine blading.

At vibrational tensile stresses of 8000 p.s.i. (3·5 t.s.i.; 5·5 kg./mm.²), damping capacities of the alloys are ten times higher than those of similar non-ferromagnetic materials. The 100-hour rupture stress at 1200°F. (650°C.) is of the order of 50,000 p.s.i. (22·5 t.s.i.; 35 kg./mm.²) and the endurance limit is in the region of 40,000 p.s.i. (18·0 t.s.i.; 28 kg./mm.²).

Optimum composition, within the range examined, is governed by the properties required in specific applications. Additions of chromium and silicon improve oxidation-resistance, but lower the Curie temperature and decrease damping capacity, and increase in strength achieved by solution hardening or precipitation hardening is also achieved at the expense of damping capacity.

(In connexion with the work reported in this paper attention is directed to *British Patent* 806,183; see p. 130.)

### Oxidation of Nickel-Iron Alloys

See abstract on p. 111.

### 'Ni-Resist': Properties and Applications

See abstract on p. 112.

### Oxidation-Resistance of Cast Irons

See abstract on p. 112.

### Influence of Curie Temperature on Rate of Oxidation of Nickel

See abstract on p. 106.

### Centrispun Nickel-containing Alloys and Steels

A. E. THORNTON: 'Recent Developments in Centrispinning High-Alloy Steels and Other Materials.' *Brit. Foundryman*, 1959, vol. 52, Feb., pp. 80-90.

The heat- and corrosion-resisting alloys and steels are, by virtue of their intrinsic properties, difficult to shape and form by conventional methods. In the production of annular components, however, increasing use is being made of the advantages offered by the centrispinning process, to solve the practical and economic problems involved. Centrifugal casting can, for example, meet the demands of the engineer for castings exhibiting freedom from physical imperfections, combined with adequate mechanical properties throughout the whole section, freedom from axial weakness, satisfactory oxidation- and corrosion-resistance, and adequate weldability. In this connexion the author outlines the properties typical of high-alloy steels and nickel-base alloys to which the centrispinning process has been successfully applied.

The four sections of the paper relate, respectively, to: (1) high-chromium steels, (2) chromium-nickel austenitic steels, (3) high-chromium low-nickel steels, (4) nickel-base alloys. Discussion relates to specific materials, the compositions of which are given. Emphasis is laid throughout on the properties obtainable in centrispun components, though each section includes a short commentary on the general characteristics of the group concerned and, where desirable, comparison is made with materials of other groups. The review also contains reference to the influence of composition on casting characteristics and, as a corollary, to operational procedure and

| Alloy | C % | Si % | Mn % | Cr % | Co % | Ti % | Al % | Cu % | Fe % | Ni % |
|---|---|---|---|---|---|---|---|---|---|---|
| 75 | 0·08 | 0·50 | 0·40 | 19·0 | — | 0·50 | 0·30 | 0·40 | 2·0 | Bal. |
| 257 | 0·10 | 0·50 | 0·40 | 19·0 | 18·0 | 1·80 | 1·0 | 0·40 | 2·0 | Bal. |

control necessary for maintenance of quality. Photographs are given of many of the components mentioned.

One of the austenitic steels discussed is of 18-8-type; the others are of higher-alloy heat-resisting grades. The centrispinning process has been applied to only two nickel-base age-hardenable alloys: for composition see table above.

These alloys are reviewed mainly in relation to their high-temperature properties and the effects, in this connexion, of centrifugal casting. Creep-resistance and resistance to segregation during casting are demonstrated, and histograms for both alloys indicate the excellent reproducibility of properties. A photograph illustrates some typical centrispun components in the two alloys.

### Stainless Steels: Properties and Uses

J. L. EVERHART: 'How to Select a Stainless Steel.' *Materials in Design Engineering*, 1959, vol. 49, Jan., pp. 95–110. *Manual No.* 155.

More than thirty wrought grades of stainless steel are designated as standard by the American Iron and Steel Institute, and apart from these many other proprietary types are available. The properties of these steels can vary considerably according to their composition and structure, and even within the limits of the three main groups (the chromium-nickel austenitic, and the high-chromium martensitic and ferritic grades) the basis compositions have frequently been modified to achieve improvements in individual properties. Obviously no one grade of wrought stainless steel is suitable for use under all the service conditions which obtain in the wide range of industries in which they find application. Selection of the most suitable steel for a specific purpose will depend mainly on the corrosive environment involved in the application, and, where the requirements are manifold, on weighing the advantages of corrosion- and heat-resistance, mechanical and physical properties, ease of fabrication, availability in the forms and sizes required, and cost considerations.

In this review the author outlines the advantages and limitations of the stainless steels in relation to each of these five factors. The article, which forms one of the 'data sheets' regularly issued in that journal, is intended as a basic guide to the engineer in selecting, from the many steels available, suitable grade for specific applications. It is restricted to discussion of the three main groups of wrought stainless steel, and in terms only of those included in the 200, 300 and 400 series of the American Iron and Steel Institute classification. Brief notes indicating the scope of each section are given below.

*Corrosion- and Heat-Resistance:* Austenitic, ferritic and martensitic types are considered in the light of their resistance to general, intergranular, galvanic and pitting corrosion. Resistance to atmospheric corrosion is discussed for normal- and high-temperature conditions.

*Mechanical and Physical Properties:* Notes are given on properties characteristic of steels of the three grades, at normal, elevated and low temperature. Textual information is supplemented by tabular matter summarizing the characteristics and main purpose of each of the A.I.S.I. grades.

*Workability:* This section includes brief reviews of hot-working, cold-working, welding and machining characteristics.

*Forms and Sizes Available:* Brief reference is made to sheet, strip, plate, tubing, bars. Mill finishes available are outlined.

*Cost Factors:* Some indication of the relative cost of the grades (based on U.S. prices) is also given.

### Solubility of Nitrogen in Austenitic Steels

F. C. LANGENBERG and M. J. DAY: 'Application of Nitrogen Solubility Data to Alloy Steel Making.' *Proc. Electric Furnace Steel Conference, Metallurgical Soc., Amer. Inst. Mining, Metallurgical and Petroleum Engineers*, 1957, vol. 15, pp. 7–15; disc., pp. 15–17.

During the war and post-war periods, in which nickel was in restricted supply, much interest developed in the austenite-promoting influence of nitrogen. This paper is, however, written in the light of the present full availability of nickel and of the resources which will in future ensure an ample and steady supply, sufficient to meet all demands.

The future of the nitrogen-containing steels depends to a large extent on the development of economic manufacturing practice, and in this connexion the authors emphasize the value of nitrogen-solubility data. Methods of predicting nitrogen-solubility are discussed and predictions so made are corroborated by reference to experimental data. The influence of temperature on solubility is considered, and the relationship between liquid and solid solubilities is briefly reviewed. Finally, the relevance of such data to the production of nitrogen-containing steels is outlined. Much of the information and data given relates to nickel-containing steels. Reference is made, for example, to experiments which show that in steel containing chromium 16, manganese 15, per cent. with nitrogen and nickel, the soundness

of the steel increases with increase of nickel content over the range 1–10 per cent. It is concluded that this improvement in soundness is probably due to the action of nickel as a strong austenite former: more specifically, nickel probably prevents the formation of excessive delta ferrite during initial solidification.

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G. R. ADAMS, W. O. BINDER and J. THOMPSON: 'Some Notes on the Nitrogen-Porosity Relations in Chromium-Nickel-Manganese Steels.'

*Ibid.*, pp. 17–20; disc., p. 20.

The nitrogen content of the low-nickel chromium-nickel-manganese austenitic steels may, if it is higher than the limits of solid solubility, contribute to ingot porosity. Solid solubility is largely a function of composition of the steel, and the purpose of the work described was to determine the effects, on the solid solubility of nitrogen, and therefore on porosity, of varying the nickel and manganese contents of such steels. (Chromium, carbon and silicon contents are not normally varied commercially and were therefore maintained at a constant level.)

Porosity was determined in steels covering the following range: carbon 0·06–0·08, silicon 0·4–0·5, chromium 17·5, manganese 7–14, nickel 0–5, per cent. The amounts of nitrogen present were varied. Approximately ninety heats of steel were cast into split iron moulds 2-in. (5-cm.) square, and then sectioned longitudinally to reveal porosity. (The hydrogen content of the heats was in the region of 6–8 p.p.m., so that any porosity observed could be attributed solely to rejection, during freezing, of nitrogen in excess of the limit of solid solubility.)

Based on the observations made, curves were prepared to show the maximum amount of nitrogen retainable, without occurrence of porosity, (*a*) in chromium-manganese steel as a function of manganese content, and (*b*) in chromium-manganese-nickel steels as a function of the nickel present.

Additional experiments were concerned with the effect, on porosity, of rate of cooling of chromium-nickel-manganese steels containing nitrogen within the range of 0·25–0·34 per cent.

The results of the respective tests confirm the influence of composition of the steel on the solubility of nitrogen. Under limited conditions, the effect, on solubility limits, of cooling rate during ingot solidification was found to be negligible.

### Influence of Stabilizing Additions on Precipitation and Intercrystalline Corrosion in Chromium-Nickel Stainless Steel

K. BUNGARDT and G. LENNARTZ: 'Influence of Stabilization on Precipitation and Intercrystalline Corrosion in a Heat-Resisting Niobium-Stabilized Steel containing 16% Cr, 13% Ni, Molybdenum and Vanadium.'

*Archiv f.d. Eisenhüttenwesen*, 1959, vol. 30, Jan., pp. 23–9.

A series of investigations, including some by the present authors (see, for example, *ibid*, 1958, vol. 29, pp. 359–64; *Nickel Bulletin*, 1958, vol. 31, No. 9–10, p. 271–2), have shown that precipitation in niobium- or titanium-stabilized chromium-nickel steels heated at various temperatures after solution-treatment occurs by formation of $Cr_{23}C_6$, re-dissolution of $Cr_{23}C_6$, and formation of the monocarbide MC, and sigma phase. Precipitation might be expected to proceed similarly in a niobium-stabilized chromium-nickel-molybdenum steel containing vanadium and nitrogen additions, but in this case two additional factors are involved: the influence, on precipitation, and thereby on susceptibility to intergranular corrosion, of the increased nitrogen content, and the degree to which vanadium will take the place of niobium in combining with nitrogen and carbon. BUNGARDT and LENNARTZ (*loc. cit.*) found that niobium nitride possesses a solubility similar to that of niobium carbide and that part of the nitrogen will combine with the niobium, to the detriment of carbon stabilization. RIEDRICH and KOCH (*Stahl und Eisen*, 1940, vol. 60, pp. 30–1) concluded that vanadium has no stabilizing effect in a straight chromium-nickel austenitic steel. These factors were further studied by the present authors.

The six steels investigated were of the following nominal composition: carbon 0·06, chromium 16, nickel 13, molybdenum 1·3, vanadium 0·7, per cent. Full details of composition, heat-treatment and test conditions are given. Three of the steels containing, respectively, 0·106, 0·137 and 0·159 per cent. of nitrogen, were used to determine the carbides and/or nitrides present in the steel in the solution-treated condition. To establish the influence of creep testing, on the precipitation reaction, specimens of the steel containing 0·159 per cent. nitrogen were heated without stress for 5000 hours at 650°C. and compared with others creep-tested to rupture, under various stresses, at the same temperature. The other three steels were used to follow the progress of precipitation occurring as a result of heating for the times and at the temperatures shown in the table below.

**Temperature and periods of re-heating of hardenable austenitic steels containing molybdenum and vanadium**

| Temperature (°C.) | Time (hours) |
|---|---|
| 450 | 500, 1,000, 3,000, 5,000, 10,000 |
| 500 | 100, 500, 1,000, 3,000, 5,000, 10,000 |
| 550 | 10, 100, 500, 1,000, 3,000, 5,000, 10,000 |
| 600 | 1, 10, 100, 500, 1,000 |
| 700 | ½, 2, 10, 100, 500 |

The steels were exposed in the following conditions: quenched; quenched and hardened; quenched and cold-worked; quenched, cold-worked and hardened. In two of the steels the niobium contents (1·32 and 1·26 per cent.) were sufficiently high to combine with the entire carbon and nitrogen contents; the niobium content of the third (0·87 per cent.) was not sufficiently high to achieve this degree of stabilization.

Precipitation behaviour was studied by chemical analysis and X-ray examination of isolated precipitate particles, and resistance to intergranular corrosion was assessed on the basis of the copper-sulphate/sulphuric-acid test.

In the quenched condition a mixed carbonitride [(Nb, V) (C, N)] was found, the vanadium content of which increased with increase in the niobium content of the steel and with rise in the temperature from which the steel was quenched. When heated at temperatures in the range 500°–700°C. specimens which contained amounts of niobium insufficient to combine with both the carbon and the nitrogen showed precipitation of chromium carbide, which contained a significant proportion of vanadium. Sigma phase was found to precipitate only after precipitation of chromium carbide was complete, and even then in amount less than that found in fully stabilized specimens. In the fully stabilized steels heating in the 500°–700°C. range produced no sign of chromium-carbide precipitation, but niobium carbonitride containing a small percentage of vanadium was precipitated and also sigma phase, the precipitation of which occurred earlier than in the insufficiently stabilized steel. No vanadium carbide or vanadium nitride was found among the precipitates.

In all cases cold-working increased the rate of precipitation and in this respect creep testing had an even greater effect, particularly on precipitation of sigma phase. Hardening (for 5 hours at 750°C.) prior to prolonged heating at 650°C. may, with certain reservations, be regarded as a preliminary stage initiating the precipitation which occurs during further heating: in a cold-worked highly stabilized steel such treatment can even result in formation of sigma.

The results of tests to determine susceptibility to intergranular corrosion may be interpreted satisfactorily in terms of chromium impoverishment. In addition, they confirm the authors' previous findings (*loc. cit.*) that account should be taken of the nitrogen content in estimating the niobium content necessary for fully effective stabilization. The solubility of vanadium in chromium carbide and its presence in the niobium carbonitride is of some value in relation to the insufficiently stabilized steels, in that the amount of chromium precipitating as carbide is thereby slightly decreased. It is considered that the amount of carbon in solution prior to heating can be correlated with subsequent susceptibility to intercrystalline corrosion and that metallographic analysis is an important tool in this respect.

**Resistance of High-Alloy Steels to Corrosion by Nitric Acid**

J. BÜNGER: 'Corrosion of High-Alloy Steels by Nitric Acid.'

*Werkstoffe und Korrosion*, 1958, vol. 9, Dec., pp. 747–55.

Much work has been carried out to investigate the resistance of various materials to corrosion by nitric acid, but mainly on an *ad hoc* basis, in relation to specific corrosion problems. Following a comprehensive series of corrosion tests, Fontana was able to evolve a diagram indicating the resistance of an 18–8-type chromium–nickel stainless steel to corrosion by the acid in a wide range of concentrations and at temperatures up to boiling point, but no iso-corrosion diagrams existed for other types of the stainless-steel series. The work recorded in the present paper was conducted to amplify information relevant to such materials.

Twenty-one steels were investigated. Sixteen were selected as representative of the straight-chromium grades and the chromium–nickel 18–8 and heat-resisting types, with or without stabilizing elements or copper or molybdenum additions: the other five, which were of straight-chromium type, were chosen to demonstrate the effects of variation in chromium and carbon contents.

Specimens were exposed to nitric-acid solutions of concentrations ranging (generally at intervals of 10 per cent.) from 1 to 67 per cent., maintained at temperatures varying, at intervals of 20°C., up to the boiling points of the solutions. Tests at boiling point lasted for 24 hours; at other temperatures they were continued for 7 days. Weight losses for each steel, converted into corrosion rates, were plotted as a function of temperature and acid concentration. The corrosion diagrams so obtained are presented and discussed in the paper.

Data derived from tests with boiling nitric-acid at concentrations of 1, 50 and 67 per cent. are used to give a graphical evaluation of the influence, on the corrosion rate, of chromium, nickel, molybdenum, silicon, manganese and copper. It was found that the minimum chromium content necessary to achieve passivation rose with increase in the carbon content and in the concentration of the acid: exposure to 67 per cent. nitric acid caused severe corrosion in 17 per cent. chromium steels, and it is recommended that for service involving contact with high concentrations of the acid this chromium content should be increased sufficiently to give a margin of safety. In concentrated acids nickel tended to increase corrosion, but in dilute acids it had no effect. In general, molybdenum did not influence corrosion-resistance, although amounts up to 2 per cent. conferred some benefit in 67 per cent. acid: with higher molybdenum corrosion increased. Silicon proved deleterious in all concentrations of the acid; manganese had an inhibiting effect, especially in the 50 per cent. acid, and a minimum manganese content of 0·70 per cent. is recommended in steels handling nitric-acid solutions. Corrosion was increased by the presence of copper.

To determine the validity of the hypothesis that at a sufficiently dilute concentration the oxidizing characteristics of the nitric acid would be too weak to induce passivation, numerous tests were carried out involving exposure for 24 hours to boiling nitric-acid solutions at concentrations varying from 0·001 to 10 *N*. Weight loss, calculated from photometric determination of the amount of iron taken into solution, was converted into a corrosion rate which is expressed graphically, as a function of concentration, for each steel.

The corrosion rate of 13 per cent. chromium steels exhibited a pronounced maximum with acid concentrations in the 0·1–1*N* range: the severity of the attack increased with increase in the carbon content. Steels containing 17 per cent. chromium suffered maximum attack in the same range only when their carbon content was relatively high. Steels containing 13·8 per cent. of chromium exhibited a peak in the corrosion curve with 0·1–1*N* solutions, but with 14·9 per cent. chromium this effect was not observed. From these and other results it is concluded that avoidance of this effect is contingent on ensuring a chromium content which, making allowance for the percentage needed for formation of chromium-iron carbide, is more than 14·5 per cent. The 20 per cent. chromium steels displayed no peak in the corrosion curve at low concentrations of acid, nor, with the exception of a sulphur-containing free-machining grade, did the austenitic chromium-nickel steels, which similarly contained the requisite amount of chromium. The hardenable grades were most resistant in the hardened condition. The results generally confirmed the high resistance of the 18-8-type austenitic steels to attack by nitric acid (corrosion rates with 1, 6 and 10*N* solutions were, respectively, 0·01, 0·1 and 0·25 mm. per year).

**Corrosion of Austenitic Steel by Nitric Acid in Presence of Chlorides**

T. E. EVANS and P. A. HAYES: 'The Effect of Chloride on the Corrosion of Austenitic Steel in Nitric Acid.' *U.K. Atomic Energy Authority, Industrial Group Report R & DB(C)TN*-124, Apr. 7, 1955; 6 pp. De-classified 1958.

The investigations described in this and the following report originated in the delivery, to the A.E.A. processing plant, Springfields, of uranium-ore concentrates which contained appreciable amounts (about 1·25 per cent.) of chlorides. Since previous experience had indicated that the presence of chlorides in the concentrate could cause corrosion of the 18-8-type titanium-stabilized stainless steel used for the plant, it was anticipated that attack on the ore-dissolving plant could be particularly severe. Experiments were therefore carried out (1) to determine the degree to which the presence of chlorides is deleterious, and (2) to establish the maximum chloride content compatible with adequate corrosion-resistance in the plant material. The results of phase one are recorded in the present report; those of phase two are contained in the report referred to in the second abstract.

The ore-dissolving process is a continuous one, in which acid and ore slurry are fed to the cascade dissolvers as the concentrated uranium-bearing solution is removed. These conditions could not be simulated in the laboratory. Specimens of titanium-stabilized 18-8 chromium-nickel stainless steel were therefore exposed (at various temperatures between 60°C. and the boiling point of the solutions) to various concentrations of nitric acid containing differing amounts of uranyl nitrate and chloride ions (in the form of sodium-chloride additions). The programme was carried out in three phases:

(1) Tests, at 60° and 80°C., on specimens immersed in nitric acid (300 g./L. $HNO_3$) containing 320 g./L. of uranyl nitrate and chloride ion in amounts up to 25·6 g./L.

(2) Tests, at 60° and 80°C., on specimens immersed in, or at 60°C. suspended in the vapour phase of two concentrations (320 g./L. and 800 g./L.) of nitric acid, containing 320 g./L. of uranyl nitrate and 6·4 g./L. of chloride ion.

(3) Tests, at 9·5°C. and boiling point, on specimens immersed in, or suspended above, solutions of nitric acid (800 g./L. $HNO_3$), with and without chloride-ion additions (6·4 g./L.).

Corrosion was found to occur mainly as a result of exposure to the vapour phase of the solutions: rate of corrosion in the liquid phase was but little affected by addition of chloride ions. Corrosion of specimens in the vapour phase is considered to be due principally to contact with condensed vapour, in which, as a result of the formation of volatile nitrosyl chloride, the concentration of chloride is likely to be higher than that of the original solution. Depending on the extent to which drops of condensate are allowed to accumulate on a particular area, presence of chlorides may, in the vapour phase, give rise either to localized pitting or to a more general surface attack. Under service conditions the former type of corrosion is potentially the more dangerous.

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T. E. EVANS and P. A. HAYES: 'Corrosion of 18-8 Austenitic Steel in Nitric-Acid Environments containing Chlorides.'

*U.K. Atomic Energy Authority, Industrial Group Report IGR-TN/C*-305, Mar. 13, 1956; 8 pp. + figs. and tables. De-classified 1958.

Following the finding that contact with the condensate from nitric-acid solutions containing up to 1 per cent. of chloride resulted in serious selective corrosion of chromium-nickel stainless steel (*see* preceding abstract), a further programme of tests was initiated with the main aim of determining the maximum chloride concentration which could be safely tolerated under any particular combination of conditions. In this connexion some of the tests

were intended to determine the composition of the condensate relative to that of the solution whence it was derived: in others the influence of temperature and acidity was studied. The tests were carried out at temperatures ranging from 40°C. to the boiling point of the solutions (about 120°C.), and at two concentrations of nitric acid, corresponding, respectively, to the composition of the acid fed to the cascade dissolvers and to that of the dissolver solution, i.e., 800 g./L. and 220 or (with boiling solutions) 100 g./L. The influence of chloride concentrations was studied over the range 0·15–10 g./L. In tests involving study of corrosion of 18–8 steel during dissolution of a chloride-containing uranium concentrate, the amount of concentrate added was designed to yield a final concentration of approximately 310 g./L. in solution, and sufficient nitric acid was added to give a final free-acid concentration of 180–210 g./L. Sodium chloride was added in amounts to give chloride contents of 0·3, 1·25 and 5 g./L. The dissolution cycle was repeated once daily, five days a week. In all the tests the specimens were suspended above the solution, in such a way as to ensure that drops of condensate would spread over the whole surface.

The results show that free acidity, chloride concentration, temperature and the presence of dissolved oxygen all play an important rôle in determining the degree to which the steel is selectively attacked. That such attack occurs only as a result of exposure to the condensate from the solution is due to the ready replenishment of dissolved oxygen, which acts as a cathodic depolarizer: in the solution itself the dissolved-oxygen content is apparently high enough to stimulate attack by chloride ion.

The influence of temperature is the reverse of that normally characteristic of corrosion reactions: selective attack is most severe at low temperature (40°C.), when the condensate, although slow to collect on the corroding surface, does not rapidly volatilize again. At higher temperatures re-evaporation of the condensate is more rapid, and attack is less severe.

Little or no attack occurs with condensate of low free-acidity (0·1–0·3*N*), originating from solutions containing about 220 g./L. of $HNO_3$. Condensates of high free-acidity (approximately 10*N*) derived from bulk solutions containing about 800 g./L. of acid may give rise to serious corrosion: under these conditions the chloride concentration conducive to selective attack decreases from 2·5 g./L. at 80°C. to 0·3125 g./L. at 40°C.

The results of analyses of the condensate showed that, at high free-acidity, chloride from the main solution becomes concentrated in the condensate: at low free-acidity chloride in the solution is much less volatile. Corrosion data obtained during dissolution of the uranium concentrate were similar to those obtained with a synthetic solution of equivalent composition.

The following methods of minimizing corrosion are recommended:

(1) Maintenance of low free-acidity (0·1–0·3*N*) in the condensate.

(2) Maintenance of low concentration of chloride, particularly if the free-acidity of the condensate is higher than the above level. With a free-acidity of 10*N* in the condensate, the chloride concentration should be below about 1 g./L. Cl in the bulk solution, otherwise severe selective attack by the condensate will occur. With this high free-acidity it may not be possible to eliminate corrosion entirely: even as little as 0·3 g./L. Cl in the bulk solution produces slight selective attack by the condensate.

(3) Ensuring, as far as possible, that condensation is prevented, particularly on low-temperature surfaces.

**Corrosion Testing by the Huey Boiling-Nitric-Acid Method: Recommended Procedure**

H. N. JONES: 'A Study of Reliable Methods of Assessment of the Corrosion-Resistance of Chromium-Nickel Austenitic Steels in Nitric Acid. A Review of Progress to 1/9/52.'

*U.K. Atomic Energy Authority, Industrial Group Report R and DB(C)TN*-16, Nov. 13, 1952; 8 pp. De-classified 1958.

Operation of the atomic energy plant at Windscale involves use of solutions which contain high percentages of nitric acid, and are maintained at elevated temperatures. It was necessary, therefore, to ensure that the 18-13-1 chromium-nickel-niobium steel used for the plant would exhibit adequate resistance under these conditions. The Huey boiling 70 per cent. nitric-acid test was used by the manufacturers to evaluate corrosion-resistance of the steel in terms of weight-loss. The penetration rates ranged, however, from a fraction of a millimetre to 26 millimetres per year, and, in view of this inordinate amount of scatter, it was decided to investigate the causes, with the aim of devising more closely controlled methods of carrying out the test and thus securing more consistent results. The work is recorded in this report.

The variables studied, the scope of the experiments and the main findings are outlined below.

*Effect of Contamination of the Test Solution*

The influence, on rate of corrosion, of the presence, in solution, of corrosion products was investigated by variation of the surface-area/solution-volume ratio or of the periods at which the acid was changed. In other experiments nitrates of iron, nickel or chromium were added to the nitric acid.

It was found that with use of the normal reflux-condenser type of apparatus such contamination considerably increased the rate of corrosion, an effect which is attributed principally to the presence of chromate ions formed in the acid from the chromic nitrate dissolved from the steel. The presence of ferric ions appeared also slightly to increase corrosion. The rate of corrosion increased in proportion to the amount of contaminating ions present and

therefore varied with the ratio of specimen-area to solution-volume and with the frequency with which the acid was changed.

The presence of chromate ions could be prevented by employing a condenser of the internal or 'cold-finger' type.

*Preparation of Specimens*

The corrosion rate for hand-polished specimens of 18-13-1 steel does not attain its true level in the boiling-nitric-acid test until after at least 800 hours of testing. In an attempt to minimize this 'incubation' period, tests were carried out on specimens the surfaces of which, prior to testing, had been pickled, electrolytically treated, or activated by heat-treatment *in vacuo*. These attempts proved unsuccessful, though the results showed that hand polishing before testing is superfluous and has no effect on the rate of corrosion.

*Statistical Analysis of the Results*

On the basis of the results obtained, a method was evolved by which the penetration rate of a rectangular specimen may be calculated. Due allowance is made for diminution of surface area and for the incubation period. The calculation therefore permits assessment of the true long-term corrosion rate.

*Alternative Methods of Measuring the Corrosion Rate*

(1) *Measurement of Dimensional Change*

Length, breadth and thickness of specimens were plotted against time and converted into penetration rates. It was found that, provided the geometry of the specimen is sufficiently uniform and pitting is not excessive, dimensional changes give as accurate an estimation of penetration rate as weight loss.

(2) *Analysis of Corroding Solution*

The results of such analysis show that iron, chromium and nickel dissolve in the same proportion as that in which they are present in the steel. Owing to the large number of determinations required, analysis of the corrodent is not expected to prove a practical method of corrosion testing.

(3) *Metallographic Examination*

It was noted that heavy pitting generally occurred at the edges of specimens, particularly those at right angles to the direction of hot rolling. Metallographic examination is considered useful only as a means of qualitative assessment of pitting, but, if used in conjunction with weight or dimensional data, it contributes considerably to the validity of the quantitative results.

On the basis of the data obtained, procedure outlined below is recommended for use in the boiling-nitric-acid testing. Specimens so tested show a standard deviation of only about 10 per cent.

'A rectangular piece is cut from the steel to be tested, and heavily etched in a boiling nitric-acid/dichromate mixture. This enables the direction of hot rolling to be distinguished. (It is obviously unnecessary with a cast steel.) Test samples are then cut from the sheet, with their length in the direction of rolling. For 350 ml. of nitric acid, the appropriate size is 1-in. × ½-in. × ¼-in. (25 × 12 × 3 mm.). (Small variations in the actual finished dimensions are not important, but the rectilinear shape of the specimens must be maintained.) The specimens are then stamped, annealed for 20 minutes at 1075°C. and water-quenched. The scale is removed by giving the specimens a few strokes with rough emery paper, and by pickling in a $HCl/HNO_3/H_2SO_4$ mixture, fol'owed by nitric acid.

'The samples are accurately measured with a micrometer and weighed to the nearest 0·01 milligram. They are then inserted into glass cradles which hold them loosely in a vertical position, and dropped into 500-ml. flasks fitted with 'cold-finger' condensers. Some pieces of crushed alumina combustion boat are also placed in the flasks to prevent bumping, and 350-ml. of acid are added to each flask, which is then boiled on a hot plate. The acid is changed every Monday, Wednesday and Friday, the extra period at week-ends making no noticeable difference to the results provided that a 'cold-finger' condenser is used. Weights and dimensions are checked weekly, and the penetration rate is calculated as described in the paper.'

**'Monel' Roofing**

See abstract on p. 111.

**Occurrence of Sigma Phase in Niobium-stabilized Austenitic-Steel Weld Metal**

C. G. MUKHIN and N. Y. PALCHUK: 'Niobium in Stainless-Steel Weld Metal.'

*Brit. Welding Jnl.*, 1959, vol. 6, Feb., pp. 82-6. Translation from *Avtomaticheskaya Svarka*, 1957, No. 2.

BAERLECKEN and HIRSCH (see abstract in *Nickel Bulletin*, 1955, vol. 28, No. 8, p. 136-7) found that when niobium-bearing 18-8-type stainless steels are subjected to prolonged heating the free niobium present, although less active in this respect than titanium, silicon, chromium or vanadium, tends to promote formation of *sigma* phase. The niobium-containing phases variously reported in the literature as being formed in austenitic chromium-nickel steels are briefly reviewed by way of introduction to the present paper. In the experiments reported, tests were conducted on samples cut from weld metal of 18-10 chromium-nickel steel of the carbon 0·1 per cent. level and containing niobium in amounts varying from 0·7 to 3·05 per cent.

Duplicate specimens were first subjected to (*a*) holding for 2½ hours at 1100°C., followed by water-quenching, or (*b*) holding at 850°C. for 3 hours, followed by air-cooling (i.e., a stabilizing-annealing treatment). Specimens in the conditions obtained

by treatments (*a*) and (*b*) were then heated at the test temperature (650°C.) for times varying up to 1000 hours.

Mechanical properties were determined on weld material in the as-welded condition and after the 650°C. exposure, and phase changes occurring on welding and heating were established by X-ray analysis.

The results indicate that alloying of 18-8-type welds with niobium lowers the ductility of the weld metal in proportion to the amount of niobium present. In welds containing more than 2 per cent. of niobium the following phases are present, in addition to the carbide of niobium: during welding, the niobide $Fe_3Nb_2$ and the silicide $Fe_4Nb_5Si_3$, and, as a result of heat-treatment under various conditions, a phase which the authors designate *H*. This is interpreted by the translator as signifying the *N* phase, which had been identified by Binder in steels containing more than 1 per cent. niobium, and was considered by him to be the carbide $M_6C$. It is stated that this phase can form in ferrite or in austenite: its presence is accompanied by loss of ductility in the weld metal. The crystal lattice of the *N* phase is isomorphous with that of the *chi* phase found in 18-8 chromium-nickel steels containing molybdenum.

Prolonged heating at 650°C. caused marked reduction in ductility: in welds containing less than 2 per cent. of niobium the change was caused by formation of *sigma* phase: in the higher-niobium welds the deterioration was attributed to formation of the *N* phase. In weld metal containing more than 2 per cent. of niobium preliminary quenching had little effect on retention of impact strength after prolonged tempering: values were in all cases low.

### Brazing of Precipitation-Hardening Stainless-Steel Sandwich Panels

A. A. LANZARA and A. M. SETAPEN: 'Sterling-plus-Lithium Alloy Selected for Brazing B-58 Stainless-Steel Sandwich Panels.'

*Welding Jnl.*, 1959, vol. 38, Feb., pp. 118-24.

This report is based on research and experience connected with development of '17-7 P.H.' chromium-nickel stainless-steel sandwich panels for the Convair B-58 aircraft.

Some 230 brazed panels are incorporated in the engine nacelles, wing-trailing edge and elevons, and the service conditions involve exposure to temperatures up to 800°F. (425°C.) from the exhaust stream, and to radiant heat from the engine. An additional 71 panels are used in the wings and control surfaces of the detachable bomb pod, where the airfoil sections are subject to severe heating due to skin friction at high speeds. The brazed sandwich panels are rated for a maximum service temperature of 900°F. (480°C.): adhesive-bonded panels used elsewhere on the aircraft have a maximum service temperature of 300°F. (150°C.). Four basic designs of brazed sandwich panel are used: wedge-shaped, flat, contoured and airfoil sections: some illustrations are shown in this article. Irrespective of shape, all sandwich panels are made up of the same types of components; essentially, each panel consists of the honey-comb-core material machined to the desired contour and depth, with a skin brazed to the upper and lower surfaces. To provide additional strength and rigidity around the edges of the panels, where most of the load is introduced, flat or angle strips, called doublers, are sandwiched between the skin and the edge-closure members. The shape of these closures is determined by the form of the panel.

All components of the honeycomb sandwich consist of '17-7 P.H.' stainless steel: design of core, surface skin, etc., is illustrated.

Among the problems which had to be solved in translating the concept of honeycomb construction into a production possibility was the development of brazing alloys capable of giving sound, strong joints capable of retaining adequate strength and corrosion-resistance under service conditions. Screening tests led to selection of a sterling silver + lithium alloy as giving the best combination of wetting and brazing characteristics, strength, and corrosion-resistance, and this is now being used by Convair. Research is still in progress with a view to further improvement of brazing alloys. Preparation and assembly methods used in brazing the stainless-steel sandwich panels are described by the author, with illustrations, and notes are added on inspection methods used on the brazed assemblies. It is noted that the development of stainless-steel sandwiches, and of practical techniques of assembling and brazing them in quantity, have made a vital contribution to the performance of the B-58. These sandwich panels have an exceptionally high strength-to-weight ratio, particularly at elevated temperatures, and, compared with conventional sheet steel and stiffener design of equal load capacity, the panels save many hundreds of pounds per aircraft.

### Welding Stainless Steel to Carbon and Low-Alloy Steels

See abstract on p. 113.

### Determination of Aluminium in High-Temperature Alloys

H. ZIBULSKY, M. F. SLOWINSKI and J. A. WHITE: 'Colorimetric Determination of Aluminium in Heat-Resistant Alloys.'

*Analytical Chemistry*, 1959, vol. 31, Feb., pp. 280-1.

The method described is based on the fact that a cupferron-chloroform separation, followed by use of complexing agents, makes possible satisfactory colour extraction of aluminium with 8-quinolinol in chloroform. Two standard curves, at different wavelengths, were produced, to provide for determination of various amounts of aluminium up to 7 per cent.

## ANALYSIS

### Ion-Exchange Procedure for Determination of Nickel

See abstract on p. 107.

### Determination of Nickel in Uranium

See abstract on p. 107.

### Analysis of Nickel-Sulphamate Plating Solutions

See abstract on p. 108.

### Determination of Aluminium in High-Temperature Alloys

See abstract on p. 124.

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## PATENTS

### Bonding of Dissimilar Materials

*British Patent* 734,275 relates to a method of bonding two strips of dissimilar metallic materials, involving heating and pressing together the surfaces to be bonded while simultaneously cooling the outer faces of the strips. This procedure ensures that, except for the thin alloy interface, the structure and properties of both materials remain substantially unchanged during bonding. The characteristics of the finished composite are therefore predictable before processing and not contingent upon a subsequent heat-treatment which, due to the dissimilarity of the two materials, is often of doubtful efficacy.

In the present patent the same technique is claimed as a means of bonding together parts of dissimilar metallic materials having a more complex shape, e.g., tubes. In this modification also use may be made of a bonding agent (e.g., nickel powder) applied to the surface before bonding: in an example cited, nickel powder is used to facilitate bonding of a copper-lead component to aluminium.

In a preferred modification, one of the parts is porous, and is impregnated with metal after bonding.

J. B. BRENNAN. *Brit. Pat.* 805,660.
(Addition to *Brit.* „ 734,275.)

### Bonding of Aluminium to Copper-Lead Alloys

Joining of aluminium-base materials to copper-lead alloys, e.g., for bearings, is achieved by (1) plating the aluminium component with a coating of nickel at least 0·0002 in. (0·005 mm.) thick, (2) tinning the surfaces of both components at 550°–700°F. (288°–370°C.) to provide means of bonding, and (3) assembling the tinned surfaces, preferably under pressure, in the same temperature range. A copper flash is preferably applied before nickel plating. The plating sequence is described.

BORG-WARNER CORPN. *Brit. Pat.* 809,470.

### Production of Supported Nickel Catalysts

The production of supported nickel catalysts by impregnation with, and subsequent reduction of, a solution of nickel formate has been hindered because, due to the low solubility of nickel formate in water, only dilute aqueous solutions could be used for the impregnation process, and the nickel content of the catalyst was therefore low. According to the present invention use is made of a solution of nickel formate in aqueous ammonia. The presence of ammonia greatly increases the solubility of the nickel formate, makes possible use of more concentrated solutions for impregnation, and thus results in production of catalysts of relatively high nickel content. Decomposition and/or reduction of the nickel-formate/ammonia compound which results from impregnation of the support is preferably effected in an atmosphere of hydrogen or in the presence of an inert gas.

E. B. MAXTED. *Brit. Pat.* 807,733.

### Nickel Catalyst in Gas-Purification

Organic sulphur compounds are removed from coal, town, water, producer or other industrial fuel gas by means of a catalyst consisting of, or containing, as the active agent, in one and the same molecule, a compound comprising (1) molybdenum or tungsten, (2) phosphorus, (3) sulphur, and (4) one of the following metals: nickel, copper, cobalt, iron, zinc or lead. The catalysts preferably consist of a phosphothiomolybdate or phosphothiotungstate of one of the last-mentioned metals. Preparation of a nickel-phosphothiomolybdate catalyst is described.

Introduction of the fourth constituent is stated to promote the activity of the catalysts.

E. B. MAXTED. *Brit. Pat.* 806,411.

### Nickel Catalyst in Production of Piperazine

Piperazine is produced, under specified temperature and pressure conditions, by a continuous process which involves passing a stream of ethylene polyamines (in particular, diethylene triamine) over a 'Raney' nickel or 'Raney' cobalt catalyst in lump form, preferably supported on an inactivated core of nickel-aluminium alloy. The process results in high yields, and the effective life of the catalyst is stated to be exceptionally long.

DOW CHEMICAL CO. *Brit. Pat.* 809,239.

### Nickel Catalyst in Production of Carbazole

Carbazole is produced, in high yields and readily purified form, by dehydrogenation of tetrahydrocarbazole at 200°–350°C. in the presence of 0·5–5 wt. per cent. of finely divided nickel and in absence of any hydrogen-absorbing material. The finely divided nickel catalyst is preferably obtained by treating nickel-aluminium or nickel-silicon alloys with alkali. The carbazole is purified by recrystallization from acetone.

BRITISH OXYGEN CO., LTD. (inventor, S. A. MILLER).
*Brit. Pat.* 806,493.

### Nickel Catalyst in Manufacture of 3 : 3 : 5-Trimethyl-Cyclohexanol

3:3:5-trimethyl-cyclohexanol exists in two isomeric forms, one of which melts at about 58°C., the other at about 37°C.

*Patent* 805,157 covers the production of 3:3:5-trimethyl-cyclohexanol containing at least 80 per cent. of the high-melting isomer. The process involves reacting isophorone or dihydroisophorone with hydrogen, under pressure and at a temperature not exceeding 100°C., in the presence of a metallic-nickel catalyst.

*Patent* 805,158 claims a process for manufacture of 3 : 3 : 5 -trimethyl - cyclohexanol containing large amounts (at least 40 per cent.) of the low-melting isomer. Such a result is difficult to achieve solely by hydrogenation over a metallic nickel catalyst. It has been found, however, that 3:3:5-trimethyl-cyclohexanol containing at least 70 per cent. of the high-melting isomer will, when maintained under pressure in contact with a metallic-nickel catalyst containing adsorbed hydrogen, undergo isomerization, to yield at least 40 per cent. of the lower-melting isomer. This finding forms the basis of the procedure described in the patent.

In both cases the metallic nickel catalyst containing adsorbed hydrogen may be of any known type, e.g., 'Raney' nickel or metal produced by reducing nickel carbonate or oxide with hydrogen.

HOWARDS OF ILFORD, LTD. (inventors, E. G. PEPPIATT and R. J. WICKER). *Brit. Pats.* 805,157. 805,158.

### Electrodeposition of Bright, Uniform, Ductile Nickel Coatings

Addition of one or more sulphonated quaternary ammonium compounds to acid plating solutions (containing, for example, sulphuric acid, fluoboric acid or boric acid), as used for deposition of nickel, copper or zinc, is claimed to result in production of brilliant, smooth, adherent, pore-free, uniform, ductile coatings, and, in this connexion, to obviate the deleterious effects (e.g., brittleness and streakiness) which have accompanied the introduction of some other additives used for the purpose of brightening and smoothing deposits.

The patent includes examples of the types of additives covered and gives solution compositions and operating data suitable for deposition of nickel, copper and zinc. Acid solutions in accordance with the invention are stated to be of particular value in plating printing rollers, embossing dies and gramophone-record matrices.

A. RIEDEL and F. BACHMANN (RIEDEL AND CO.) (inventor, G. MICHAEL). *Brit. Pat.* 805,316.
Similar to *French* „ 1,155,823.

### Electrodeposition of Bright Coatings from Plating Solutions containing Inorganic Impurities

Addition, to bright-plating solutions, of one or more tertiary amino compounds containing no neutralized or un-neutralized carboxyl groups is claimed to nullify the adverse effects of any inorganic impurities present in the electrolyte, and thereby to ensure the effective functioning of the brightening agents. Classes of compounds falling within the scope of the invention are defined and examples are given.

The additives may be used in electrodeposition of bright coatings of copper, nickel, zinc, chromium and precious metals.

DEHYDAG DEUTSCHE HYDRIERWERKE G.m.b·H.
*Brit. Pat.* 806,403.
Similar to *German* „ 934,508.

### Treatment of Copper-Nickel Alloys prior to Silver Plating

Copper or copper-base alloys are cleaned, preparatory to silver plating, by pickling in an aqueous solution of an alkali, amine or ammonium salt of thioglycollic acid. Preferably the ammonium thioglycollate is equivalent to 5-15 per cent. w./v. thioglycollic acid. Addition of silver (preferably in the form of silver acetate), to give a maximum silver content of 2 per cent. w./v. in the solution, will ensure not only adequate cleaning but also deposition, on the copper or copper alloy, of a thin adherent film of silver. This result has the effect of obviating the need for a silver flash before electroplating, as, for example, is demonstrated by reference to the application of the treatment to plating of nickel silver.

ROBINSON BROS., LTD. (inventor, S. A. M. THOMPSON).
*Brit. Pat.* 807,172.

### Nickel Plating of Aluminium, Titanium, Zinc and Lead

Adherent electrodeposits of chromium, nickel, copper or zinc are obtained on aluminium, titanium, zinc or lead articles by exposing the article, prior to plating, at room temperature, to the action of hydrogen-chloride gas until it appears to sweat.

P. J. TOPELIAN, assignor to TIARCO CORPN.
*U.S. Pat.* 2,856,333.

### Treatment of Uranium Preparatory to Nickel Plating

Difficulties have been experienced in using conventional surface-treatment procedures to prepare uranium for electroplating. According to the present invention, uranium may be satisfactorily electroplated or dip-coated by treating it, immediately prior to coating, with concentrated hydrochloric acid sufficiently long to form a black layer, which is then dissolved off by concentrated nitric acid. Details are given of procedures suitable for plating uranium with nickel, iron or zinc.

U.K. ATOMIC ENERGY AUTHORITY. *Brit. Pat.* 805,781.

### Electroless Nickel-Plating Solutions of High Stability

Nickel/hypophosphite electroless-plating solutions suffer from two disadvantages: rates of deposition

of metal are comparatively slow and the solutions have a tendency to decompose, with formation of a precipitate, resulting from uncontrolled reduction of nickel. Use of higher operating temperatures increases the rate of deposition but favours decomposition of the electrolyte.

According to the present invention this problem is resolved by addition, to the nickel/hypophosphite solution, of a compound of molybdenum, in an amount such as to inhibit decomposition without affecting the plating reaction. The molybdenum compound (preferably molybdic acid, added as such or viâ molybdic anhydride) is calculated to give a molybdic-acid content not exceeding 0·035 g./L. The solution, which should contain 1–15 g./L. of nickel ion and 2–40 g./L. of hypophosphite ion, is operated at 150°–212°F. (65°–100°C.) and an initial pH value of 4·5–6. Examples are given of typical electrolytes: one of the preferred compositions is as follows (g./L.): nickel sulphate 16, sodium hypophosphite 18, propionic acid 10·4, hydroxylamine sulphate 0·6, molybdic acid 0·015. The pH is 5·2–5·6.

GENERAL MOTORS CORPN., assignee of H. J. JENDRZYNSKI. *Brit. Pat.* 806,494.

### Regeneration of Nickel Electroless-Plating Solutions

Solutions containing nickel citrate and sodium hypophosphite are regenerated by being passed through a nickel-charged cation-exchange column where calcium and cadmium ions are removed and the sodium and hydrogen ions, formed during plating by depletion of the nickel ions, are exchanged for nickel ions. The resultant nickel-citrate solution is re-circulated and the ratio of equivalents of citrate to nickel is thereby maintained at 1 : 2.

A claim is also made for regeneration of electroless cobalt-plating solutions by the same method.

S. A. HAYS, assignor to NORTH AMERICAN AVIATION, INC. *U.S. Pat.* 2,871,142.

### Electroless Nickel Plating of Mild Steel prior to Enamelling

Mild steel in sheet, strip or coil form is subjected, prior to fabrication and subsequent vitreous enamelling, to a preparatory treatment involving the following stages: (1) Cleaning by vapour blasting and/or electrolytic methods to a given degree of surface roughness ; (2) coating, from a plating solution of the nickel- or cobalt-cation/hypophosphite-anion type, with cobalt or nickel to a weight of between 50 and 170 mg./sq. ft.; (3) increasing the activity of the cobalt or nickel coating by oxidizing, and subsequently reducing the surface layer of the coating in hydrogen, in both cases at a temperature in the range 500°–1100°C.

To avoid damage to the nickel or cobalt coating in subsequent handling or forming, the deposits are protected with a removable organic coating or a thin layer of zinc, cadmium or iron. Where the protective coating is metallic it may be removed, prior to enamelling, by treatment with dilute hydrochloric acid at 100°–140°F. (38°–60°C.).

An advantage of the process, apart from the considerable improvement achieved in the enamelling characteristics of the mild steel, is that it eliminates the necessity for nitric-acid pickling.

W. H. F. TICKLE. *Brit. Pat.* 806,405.

### Electroless Nickel Plating in Production of Printed Circuits

A printed circuit is produced on an insulating base of a resin-bonded fibrous material by a process which involves the following stages:

(1) Mechanically roughening the surface of the insulating base.

(2) Activating all or part of the roughened surface by treatment with a solution containing a soluble salt of a noble metal (preferably an aqueous solution of palladium chloride containing a wetting solution). (Parts of the base on which no conductive layer is required may be masked, preferably by a nitrocellulose lacquer applied by a printing process.)

(3) Treating the activated surface with a reducing agent which precipitates the noble metal.

(4) Plating the activated portions of the base with nickel from a hot solution containing a soluble nickel salt, hypophosphite ions and a suitable buffer. (Alternatively, where the entire surface of the base has been activated, a thin coating of nickel is deposited over the surface. Parts not intended to be conductive are then covered with resist material, a thicker coating of nickel is applied to the exposed parts, and finally the resist material and the thin layer of nickel underlying it are removed.) A final layer of an alloy or another metal (e.g., a solder) may be applied to the nickel coating.

BRITISH INSULATED CALLENDER'S CABLES, LTD. (inventors, J. G. BRAZIER, E. H. REYNOLDS and W. A. WALKER). *Brit. Pat.* 806,977.

### Electroless Nickel Plating of Surgical Needles

Carbon-steel surgical needles to which are swaged a flexible surgical suture are provided with a shiny, corrosion-resistant, ductile nickel/nickel-phosphide coating deposited by the electroless process. The needles so treated can be shaped and flanged on the gut, stored in air or in conditioning fluids, and sterilized without deleterious effects.

R. M. BANKS and R. C. BELL, assignors to AMERICAN CYANAMID CO. *U.S. Pat.* 2,865,375.

### Non-Electrolytic Plating of Metals with Nickel

A metallic surface (e.g., a silver mirror) is coated with a metal (e.g., nickel, copper or cobalt) by bringing it into contact with a metal-salt solution and a metal-powder suspension, to which suspension

surface-active agents have been added. Examples of such agents are the alkyl aryl sulphonates and their sodium salts, and poly aryl sulphonates.

B. F. SCHWEIG. *Brit. Pat.* 807,975.

### Deposition of Bright Nickel by Gas Plating

Smooth, bright metal deposits are obtained by thermal decomposition of an appropriate gaseous metal compound in the presence, as a brightener, of one of the following nitrogen-containing compounds: ammonia, methenamine, nitrous oxide, ammonium hydroxide. The gaseous metal compound is preferably a carbonyl of nickel, chromium or molybdenum. A specific application is in the production of reflective coatings of nickel on rayon, nylon or other synthetic fibres. Nickel and other coatings produced by the process may be subsequently electroplated with copper without the need for first abrading the surface.

L. J. NOVAK and H. J. HOMER, assignors to COMMONWEALTH ENGINEERING CO. OF OHIO. *U.S. Pat.* 2,859,132.

### Nickel Coating Organic Fibres by Gas Plating

Immediately after heating to drive off the volatile organic solvent in which it was dissolved prior to extrusion, freshly extruded organic fibre is coated with metal by exposure to a gaseous metal carbonyl, at a temperature in the range 250°–450°F. (121°–232°C.), to achieve decomposition of the gas, and resulting deposition of the metallic constituent. Gas plating in this manner may be effected using carbonyls of one or more of the following metals: nickel, iron, chromium, molybdenum, cobalt, tin.

F. E. DRUMMOND, assignor to COMMONWEALTH ENGINEERING CO. OF OHIO. *U.S. Pat.* 2,862,783.

### Nickel-containing Coatings on Refractory Materials for Soldering

An adherent coating capable of bonding with both hard and soft types of solder is produced on non-metallic refractory components by application (and subsequent firing in a reducing atmosphere) of a composition consisting essentially of powders of the following materials: molybdenum or tungsten 5–30, nickel, cobalt or iron 40–85, manganese 35 max., glass 5–30, per cent.

D. W. LUKS and J. POWELL, assignors to FRENCHTOWN PORCELAIN CO. *U.S. Pat.* 2,857,664.

### Nickel-Manganese Alloy Resistant to Cathode Stripping

The core of a thermionic cathode consists of a nickel-manganese alloy in which the nickel content is not less than 98 (preferably 98·75) per cent., with manganese 0·2–2 (preferably 1) per cent. Impurity content is limited to 0·25 per cent. max. The relatively high manganese content is claimed to eliminate susceptibility to cathode stripping (separation of the electron-emissive oxide coating from the core), without impairing the electronic characteristics of the cathode. The cathode is of high efficiency and has long operating life.

GENERAL ELECTRIC CO. *Brit. Pat.* 805,229.

### Nickel-containing Barium Alloy for Use in Electron-Discharge Devices

Barium alloys claimed to be stable in air and capable of liberating elemental barium when heated *in vacuo* are produced by heating, in an atmosphere of hydrogen at about 800°C., a mixture of powders of barium oxide and of nickel, cobalt or a nickel-tungsten alloy. Homogeneous alloys containing up to 65 per cent. of barium are obtainable. Such alloys are particularly suitable for use as contact getters or as additions to the emission layer of the oxide cathode of electron-discharge devices, since they do not form reaction products deleterious to the satisfactory operation of the device.

TELEFUNKEN G.m.b.H. *Brit. Pat.* 805,880.

### Hot Cathode for Thermionic Tubes

A hot cathode for use in thermionic tubes at temperatures below 1000°C. is produced by application of an alkaline-earth-metal compound (preferably a mixed oxide) to a metal carrier (at least the surface of which is nickel) and then heating the compound, a portion at a time, in an inert atmosphere, to a temperature of at least 3500°C., preferably using the cathode itself as one electrode of an electric arc.

Cathodes produced in this way are claimed to possess the following advantages: emission on a par with that of an oxide-coated cathode; absence of a destructible semi-conductor coating; freedom from oxides and therefore from the risk of formation, during operation, of an intermediate layer which would reduce loadability; negligible sensitivity to contamination by impurities; negligible evaporation of metal; sensitivity to ion bombardment not greater than that of a pure-metal cathode. An additional benefit is that activation is unnecessary.

'PATELHOLD' PATENTVERWERTUNGS-UND ELEKTRO-HOLDING A.G. *Brit. Pat.* 806,032.

### Nickel-base Alloy for Spark-Plug Electrodes

Alloys displaying high resistance to erosion by electrical discharge, and to inter- or intra-granular attack when exposed to the temperatures and corrosive conditions involved in operation or fabrication of spark-plug electrodes, fall within the following range of composition: nickel 90 min., manganese 0·5–3, zirconium 0·1–2, chromium 0·5–3, silicon 0·2–1·5, iron 0·5 max., carbon 0·05 max., impurities 0·25 max., per cent. The alloys may also contain up to 1 per cent. of niobium and/or up to 0·4 per cent. of titanium, in which case the carbon content should exceed neither 0·1 per cent. nor the sum of one-fifth of the niobium content and one-tenth of the titanium content.

A preferred alloy contains: nickel 94·68 min., manganese 1·9, zirconium 0·15, chromium 1·75, silicon 0·4, titanium or niobium 0·3, iron 0·5 max., carbon 0·05 max., impurities 0·25 max., per cent.

CHAMPION SPARK PLUG CO. (inventors, R. C. TEASEL and J. F. HOFFMAN). *Brit. Pat.* 808,170.

**Nickel-containing Gas-Diffusion Electrode**

A porous double-skeleton material of high mechanical strength and high electronic and thermal conductivity, suitable for use as a diffusion electrode in a fuel cell, consists of a carrier skeleton of an electronically conductive material in which is embedded 'Raney' metal, in amount comprising 20–80 wt. per cent. of the whole. The 'Raney' metal and carrier skeleton are preferably nickel. Procedure for manufacturing the double-skeleton material is also claimed.

A hydrogen or negative electrode made from the double-skeleton material is stated to permit operation of the fuel cell at a low temperature and slightly superatmospheric gas pressure, to have a long life and high resistance to poisoning, and to ensure, without recourse to noble metals, high-efficiency operation of the cell.

RUHRCHEMIE A.G. and STEINKOHLEN-ELEKTRIZITÄTS A.G. *Brit. Pat.* 806,644.

**Production of Nickel-containing Electron-Emissive Coatings**

A nickel-aluminium alloy containing 10 per cent. of aluminium is added to an aqueous suspension containing at least one alkaline-earth-metal carbonate, by grinding the suspension in a ball mill lined with nickel, using grinding media composed of the alloy itself. During grinding the alloy is worn away and is thus incorporated in the suspension. The invention is used in preparation of electron-emissive coatings in which the active alloy is incorporated without simultaneous introduction of contaminants.

L. W. KATES, assignor to SYLVANIA ELECTRIC PRODUCTS, INC. *U.S. Pat.* 2,857,289.

**Sintered Copper-base Bearing Dispersion-Hardened with Nickel-Titanium-Alloy Particles**

The sintered bearing is produced from a mixture of metal powders comprising tin, nickel and dispersed hard particles of nickel-titanium alloy, remainder copper or copper-base alloy. Introduction of the nickel-titanium (nickel 35–85, titanium 65–15, per cent.) particles is stated greatly to increase the wear-resistance, while the bearing has the further advantage of being manufactured to close dimensional tolerances. A bearing having high oil-retaining, wear-resistant and anti-score properties is produced from a powder mixture consisting of tin 1–13, nickel 2–15, nickel-titanium alloy 2–8, carbon 0·3–4, lubricant 0·3–2 per cent., balance copper.

R. F. THOMSON, assignor to GENERAL MOTORS CORPN. *U.S. Pat.* 2,849,789.

**Nickel-Copper-Alloy in Submarine-Cable Joints**

The patent relates to the production, in single-core gas-filled submarine power cables, of joints of a construction designed to confer adequate resistance to the high internal and external pressures involved in submarine service, while overcoming difficulties resulting from induced currents in the reinforcement and armouring. To this end the jointed and re-insulated cable is enclosed in a seamless tube of high tensile strength (preferably made from an alloy containing nickel 68, copper 29, iron, manganese and carbon 3, per cent., e.g., 'Monel'), which is sealed at each end to the cable sheath. In the clearance between the tube and the re-insulated conductor are two or more strips of metal of high electrical conductivity (preferably copper) which enclose the conductor, and the ends of which are mechanically and electrically connected to the cable sheath.

BRITISH INSULATED CALLENDER'S (SUBMARINE CABLES), LTD. (inventors, E. L. DAVEY, J. CHESWORTH, W. Y. MURRAY and C. L. COY). *Brit. Pat.* 806,522.

**Nickel-Copper-Silicon Casting Alloy containing Spheroidal Graphite**

Casting alloys exhibiting good machinability, in combination with satisfactory gall-resistance, wear-resistance and mechanical properties, are produced by first treating a melt of the following composition with 1–2 per cent. of calcium carbide, and then introducing 0·1–0·2 per cent. of magnesium into the treated melt. *Composition of melt:* copper 25–35, silicon 0·5–3·5, iron 0–5, manganese 0–2·5, cobalt 0–2, chromium 0–1, carbon 0·4–0·7, per cent., balance nickel. The cast alloy has a microstructure containing nodules of graphite having a feathery edge. Compositional limits of the final alloy are the same as those for the melt, but with the addition of magnesium in the range 0·02–0·2 per cent. and calcium in amounts up to 0·01 per cent.

The patent includes discussion of the rôle of the constituent elements of the alloys and gives data illustrating the properties obtainable.

MOND NICKEL CO., LTD. *Brit. Pat.* 807,957.

**Nickel Alloy in Thermally-Compensated Magnetic Apparatus**

The performance of magnetic tachometers, moving-coil galvanometric devices and similar apparatus may be seriously affected by variations in the torque generated, as a function of temperature, by the magnet action on the movable assembly. The present invention compensates for such thermally induced variations by use, for the spiral spring, of a metal having a temperature coefficient of elasticity substantially equal to, and of a direction opposite to, that of the temperature coefficient of resistivity of the non-magnetic metal members of the movable assembly. The spring is preferably of phosphor-bronze or beryllium-bronze, and, in the latter case, the non-magnetic parts of the movable assembly may consist of a copper-nickel alloy having a nickel content of 15·07 per cent.

ETABLS. E. JAEGER. *Brit. Pat.* 805,894.

### Sintered Wear-Resistant Aluminium-base Alloys

*British Patent* 730,164 covered the production, by powder metallurgy, of copper- or ferrous-base alloys containing 0·3–3·3 per cent. carbon and 0·5–15 per cent. of a nickel-titanium alloy consisting of 35–85 per cent. of nickel and 15–65 per cent. of titanium. The present patent extends the applications of the nickel-titanium alloy to powder-metallurgical production of wear-resistant aluminium-base alloys for use in components such as bearings, piston rings, etc. These alloys contain 0·5–15 per cent. of the nickel-titanium alloy (at least 70 per cent. of which consists of $Ni_3Ti$) and may, in addition to minor amounts of other elements introduced to improve strength or anti-frictional characteristics, contain specified percentages of silicon, cadmium or carbon. Method of making a wear-resistant article from these components is also claimed.

GENERAL MOTORS CORPN. (inventor, A. L. BOEGEHOLD). *Brit. Pat.* 805,100.
(Addition to *Brit. Pat.* 730,164).

### Spheroidal-Graphite Cast Iron in Crushing Elements

Elements of crushing machinery consist of martensitic cast iron presenting a definite chill and having a hard, wear-resistant face and a tough, ductile cast iron backing containing spheroidal graphite. Compositional limits of the cast iron are as follows: carbon 3·4–4·2, silicon 0·6–1·1, manganese 0·1–0·6, chromium 0·4–1, nickel 3·5–5, per cent., together with sufficient magnesium (not exceeding 0·1 per cent.) to produce a spheroidal-graphite structure in the backing. Before casting it is essential to inoculate the molten metal with 0·2–0·6 per cent. of its weight of silicon (as such or in the form of a compound), to prevent formation of an undesirable carbide-network structure in the backing.

The characteristics of such castings render them extremely suitable for use as crushing elements. The chilled face is very hard (generally at least 600 Brinell), the chill is definite, and the spheroidal-graphite backing usually has a tensile strength of at least 31 t.s.i. (49 kg./mm.²). Such properties are obtainable in chill-cast elements having section thicknesses of 3–12 in. (7·5–30 cm.).

MOND NICKEL CO., LTD. *Brit. Pat.* 805,266.
Similar to *U.S.* „ 2,771,358.

### Nickel-Chromium-Molybdenum High-Strength Steel

Structural steels, claimed to exhibit high strength and hardness combined with good ductility and toughness when heat-treated by quenching from above the upper critical temperature and double-tempering in the range 400°–500°F. (205°–260°C.), fall within the following limits of composition: carbon 0·35–0·45, silicon 1·3–2, manganese 0·5–1·5, nickel 1·5–2·5, chromium 0·7–0·9, molybdenum 0·2–0·4, titanium 0·04–0·2, boron 0–0·0025, per cent.

S. J. ROSENBERG and C. R. IRISH, assignors to the U.S. SECRETARY OF THE NAVY. *U.S. Pat.* 2,863,763.

### Dispersion-Hardened Nickel-base High-Temperature Alloys

Materials exhibiting sustained resistance to creep at temperatures in the range 800°–1050°C. are claimed to result from compacting, sintering and working powder mixtures consisting of (1) 5–20 vol. per cent. of an insoluble compound comprising a carbide, boride, silicide or nitride of titanium, zirconium, niobium, tantalum, vanadium or hafnium, or a disilicide of molybdenum or tungsten; (2) a ductile heat-resisting matrix alloy, preferably containing (*a*) nickel 80, chromium 20, wt. per cent., or (*b*) nickel 90 max. and/or cobalt 70 max. (nickel+cobalt at least 40), chromium 5–32, iron 25 max., wt. per cent. The preferred constituent in group (1) is titanium carbide.

The sintered product should have a microstructure comprising a random dispersion of the finely divided compound throughout the metallic matrix, the maximum distance between the particles being one micron. Sintering is effected at a temperature of at least 1100°C. and the sintered body is cold-worked to achieve a reduction of at least 50 per cent.

SINTERCAST CORPN. *Brit. Pat.* 807,093.
Similar to *Canad.* „ 559,409.

### Nickel-Chromium-Iron Heat-Resisting Alloys

Nickel-chromium-iron alloys of the compositions shown below are claimed to have a tensile strength greater than 6000 p.s.i. (4·2/kg./mm.²) at 2300°F. (1260°C.), good oxidation-resistance at 2200°F. (1205°C.), 100-hour rupture strength higher than 200 p.s.i. at 2200°F. (1260°C.), and a creep rate of less than 1 per cent. when subjected to 70 p.s.i. at 2200°F. (1205°C.) for 1000 hours. Composition: carbon 0·45–0·55, nickel 34–37, chromium 23–27, molybdenum 1–2, silicon 1–2·5, manganese 1–4, aluminium 0·05–0·4, nitrogen 0·15–0·4, boron 0·001–0·04, sulphur <0·04, phosphorus <0·04, per cent., balance iron.

E. M. ANGER, assignor to DURALOY CO. *U.S. Pat.* 2,857,266.

### Cobalt-Nickel-base High-Damping Turbine-Blading Alloys

Alloys possessing high damping capacity and strength, claimed to be particularly suitable for use in turbine blading operating in the range 1100°–1300°F. (595°–705°C.), fall within the following limits of composition: cobalt 65–88, titanium 1–3, aluminium 0·1–1·8, carbon <0·1, impurities 1 max., per cent., nickel balance (at least 8 per cent.). The alloy may also contain minor amounts of other elements. The combined aluminium+titanium content should be 1·5–2·5 per cent.

The components are subjected to a solution-treatment at a temperature above 1600°F. (870°C.) and are then aged in the range 1200°–1400°F. (650°–760°C.) for at least 4 hours, to give a hardness of 250–330 VHN

WESTINGHOUSE ELECTRIC INTERNATIONAL CO. *Brit. Pat.* 806,183.

(The work on which this patent claim is based is reported in a paper abstracted on p. 117.)

### Boron-containing Titanium-base Alloy for Addition to Heat-Resisting Steels

The addition alloy of the invention provides a means of introducing into a steel from 2 to 50 p.p.m. of boron. Increase in stress-rupture properties achieved in nickel-chromium heat-resisting steels by such additions are tabulated and steels manufactured by addition of the alloy are claimed. The alloy comprises: titanium 61–90, boron 0·005–0·5, per cent., balance iron. Aluminium may be present in amounts up to 5 per cent.

UNION CARBIDE CORPN., assignee of E. H. WYCHE. *Brit. Pat.* 808,270.

### Production of Cooling Passages in Nickel-Chromium-base Turbine Blades

The usual method of removing the filler used to ensure retention of cooling holes in turbine blades during extrusion or other working to aerofoil shape is by leaching with an acid solution which attacks the filler but not the metal of the blade. In the case of blades made from heat-resisting alloys of nickel-chromium or nickel-chromium-cobalt base, fillers are usually of mild steel, iron-manganese-titanium alloys or sintered compositions of iron and magnesia. The leaching process is slow, due mainly to the resistance exhibited, in the initial stages, by the oxide layer which forms on the exposed surface of the filler during working.

According to the invention the time required for removal of filler is shortened by etching, before the preferential leaching stage, in an aqueous acid solution (preferably boiling hydrochloric acid), which attacks both the filler and the blade material. Treatment is limited to a period which will remove only the oxide film.

HENRY WIGGIN AND CO. LTD. (inventor, B. KEEGAN). *Brit. Pat.* 806,479.

### Heat-Resisting Alloy for Use in Salt Pots

Addition of small amounts of nitrogen and selenium to an iron-nickel-chromium-niobium heat-resisting alloy commonly used for cast neutral-salt pots has been found greatly to increase service life, e.g., 100 hours to 600–800 hours. Alloys in which the advantages of nitrogen and selenium additions are obtained are covered by the claims of the patent, within the following limits of composition: nickel 32–38, chromium 14–20, niobium 1–3, silicon 1–2, carbon 1·5 max., nitrogen 0·05–0·2, selenium 0·01–0·1, per cent., balance iron. Copper 0·1–1·5 per cent., and/or manganese 1–2 per cent., may also be present.

BRITISH DRIVER-HARRIS CO., LTD. (inventor, P. R. MARSH). *Brit. Pat.* 805,187.

### Forgeable Stainless Steel Resistant to Mineral Acids

Some of the steels most resistant to attack by mineral acids are suitable for production in the cast form only. According to the present invention steels falling within the following composition range shown are readily forgeable and also highly resistant to corrosion by mineral acids (exhibiting, for example, in a boiling 30 per cent. sulphuric acid, a penetration rate of less than 0·006 in./mth.; 0·15 mm./mth.). Composition: carbon 0·01–1·0, silicon 0·75 max., manganese 0·15–2, sulphur 0·04 max., phosphorus 0·04 max., nickel 23–27, chromium 17–20, molybdenum 0·8–1·5, copper 0·8–1·5, titanium 0·2–2, per cent.

R. SMITH and C. O. TARR, assignors to U.S. STEEL CORPN. *U.S. Pat.* 2,865,741.

### Manganese-Nickel Precipitation-Hardening Austenitic Steels

The invention covers manganese-nickel steels, with or without chromium, which are stated to possess good hot- and cold-working characteristics. The steels are rendered age-hardenable by addition of vanadium and appropriate adjustment of the carbon content, and the age-hardening effect may be intensified by addition of aluminium in critical amounts.

Nickel is present up to 12 per cent., i.e., in amounts sufficient, in conjunction with the manganese content, to ensure an austenitic structure. (No specific manganese content is claimed, but reference is made in the preamble to percentages up to 16.)

J. J. HEGER, J. M. HODGE and R. SMITH, assignors to U.S. STEEL CORPN. *U.S. Pat.* 2,865,740.

### Chromium-Nickel Stainless Steels Precipitation-Hardened with Molybdenum and Silicon

Stainless steels of 18–8 chromium-nickel type are rendered age-hardenable by additions of 3–5 per cent. of molybdenum and 2·5–4 per cent. of silicon. Carbon should not exceed 0·08 per cent. and chromium and nickel contents are in the range 18–25 and 8–12, per cent., respectively. Steels age-hardened in this way are claimed to exhibit high resistance to corrosion, abrasion and erosion and to possess sufficient ductility for use in applications involving exposure to stress.

N. S. MOTT, assignor to COOPER ALLOY CORPN. *U.S. Pat.* 2,861,883.

### Prevention of 'Hot-Shortness' in Austenitic Stainless Steels

'Hot-shortness' in cast (chromium-nickel) austenitic stainless steels is prevented by addition to the melt of 0·05–0·25 per cent. of vanadium and 0·01–0·15 per cent. of titanium, preferably in the form of a ferro compound. Boron, in amounts up to 0·002 per cent., is also beneficial.

The addition may be effected to the molten steel while in the furnace before, or in the ladle during, tapping. The patent includes procedures and results for a variety of chromium-nickel stainless steels.

L. E. FINK, assignor to ELECTRIC STEEL FOUNDRY CO. *U.S. Pat.* 2,867,533.

### Surface Treatment of Chromium-Nickel Alloys and Steels before Cold-Working

Surfaces of metal articles which are to be cold-worked are treated with an acid oxalate coating

material in which is incorporated a lubricant, thus combining what has been a multi-step process into one operation. The lubricating constituent (present in amounts of 0·1 to 8 per cent.) consists of one or more saturated straight-chain fatty acids having at least 12 carbon atoms (preferably stearic, myristic, lauric or palmitic acid) and/or one or more glycerides of these acids.

The process is applicable for treatment, *inter alia*, of stainless steels and nickel-chromium alloys.

AMCHEM PRODUCTS, INC. *Brit. Pat.* 807,660.

### Cold-Rolling of Small-Diameter Thin-Wall Steel Tubes

The production of small-diameter steel tubes to precise dimensional requirements has hitherto involved subjecting hot-rolled tubes to cold-drawing, or, in the case of steels which are difficult to deform, use of the Pilger step-cold-rolling process. Cold-drawing is difficult, expensive and relatively slow (drawing speeds are in the range 40–80 metres/minute), while with the Pilger step-rolling process a length of only a few millimetres can be rolled at one time, and tube manufacture is correspondingly time-consuming and expensive.

The cold-rolling process described in the patent uses rolling speeds in excess of 120 m./min., and the cross-sectional reduction per pass is equivalent to that obtained by hot rolling (in the region of 70 per cent.). It can be used to cold-roll hot-rolled steel tubes, in a continuous pass, to precision-finished components with an outer diameter of the order of 10 mm. and a wall thickness of less than 1 mm., i.e., to dimensions and tolerances hitherto achieved only by cold-drawing. Full details (with drawings) are given of the rolling equipment and of the function of the individual roll sets. The suitability of the process for cold-rolling of chromium-nickel steel tubes is specifically suggested.

DELTA-KÜHLSCHRANK G.m.b.H. *Brit. Pat.* 805,598.

### Austenitic-Steel Weld Metal with Increased Resistance to Hot Cracking

Susceptibility to hot cracking of weld deposits made, under restraint, from austenitic chromium-nickel stainless steel is minimized by introduction of about 0·1–0·25 per cent. of nitrogen into the weld metal. The nitrogen addition is preferably made by use of a welding rod of the following composition: carbon 0·01–0·09, silicon up to 1·5, manganese up to 2, chromium 17–20, nickel 10·5–12·5, niobium up to 2, nitrogen 0·1–0·25, per cent., balance iron.

R. A. PERKINS and W. O. BINDER, assignors to UNION CARBIDE CORPN. *U.S. Pat.* 2,871,118.

### Cladding Molybdenum with Nickel-containing Materials

The surfaces of molybdenum plate and of the cladding material are cleaned, and the cladding is tightly wrapped round the molybdenum, so that it slightly overlaps itself in the direction of wrapping and also protrudes over the ends of the plate. The projecting portions of the cladding are peened over the ends of the plate so that they completely cover the surfaces, and the cladding is finally bonded to the basis metal by heating, in a non-oxidizing atmosphere, to a temperature in the range 1000°–1150°C. and rolling, again in a non-oxidizing atmosphere, to a 15–40 per cent. reduction. Subsequent passes (each of 15–40 per cent.) may be effected in air, with intermediate annealing. Reductions are repeated until a thickness of about 0·09 in. (2·25 mm.) is obtained. The composite is then cleaned and finally annealed, in a non-oxidizing atmosphere, at 850°–1100°C. for 3 minutes-3 hours, according to temperature used. (Details are also given of stages which may be used to effect further reductions.)

The following materials are claimed as suitable for use as cladding: nickel; alloys containing iron 55, nickel 29, cobalt 16, per cent. or nickel 76, chromium 15, iron 9, per cent.; an 80–20 nickel-chromium alloy. Since alloys containing chromium do not readily bond with molybdenum, they are, according to this invention, plated with nickel prior to cladding.

W. M. FRASER and W. E. BRUSH, assignors to WESTINGHOUSE ELECTRIC CORPN. *U.S. Pat.* 2, 871,150.

### Nickel-Chromium-Boron/Stainless-Steel Surfacing Material

Hard-surfacing materials comprise a powder mixture of a hard alloy (essentially nickel, chromium and boron) and a material which is relatively soft, e.g., stainless steel or other material based on nickel, cobalt or iron. The preferred composition of the hard alloy is nickel 65–85, chromium 8–18, boron 2–4·5, iron 1·25–5·5, silicon 1·25–5·5, cobalt up to 1·5, carbon 0·3–1, per cent.

The powder mixture is sprayed onto the surface of the basis metal at a temperature in the plastic range and below the melting point of either constituent of the mixture. Coatings so produced are porous and oil-retaining, machinable and corrosion-resistant. They exhibit good wear- and abrasion-resistance.

W. S. BOWLES, assignor to WALL COLMONOY CORPN. *U.S. Pat.* 2,868,667.

### Nickel-base Corrosion- and Abrasion-Resistant Facing Alloys

Facing alloys which can be applied to basis metals by a low-temperature metallizing operation and subsequently torch-heated to produce a dense smooth layer are of the following composition: chromium 10–25, silicon 5–20, boron 0·25–4, copper up to 2, molybdenum up to 4, carbon 0·25 max., iron less than 5, iron + impurities less than 8, per cent., balance nickel. Application of such alloys, by the technique described, is stated to obviate certain disadvantages which are associated with facing by welding techniques.

J. W. FOREMAN, assignor to DURIRON CO., INC. *U.S. Pat.* 2,864,696.

### Coating of Nickel-base-alloy Turbine Blades with Aluminium

Gas-turbine blades fabricated from nickel-base high-temperature alloys are protected against oxide penetration during service by coating with aluminium by the method described.

The surface is treated by application of an aluminium coating produced either (*a*) by removing surface oxide by dipping the component into a fused-salt flux containing potassium chloride, sodium chloride, cryolite and aluminium fluoride, and then dipping it in molten aluminium; or (*b*) by spraying the component with molten aluminium and then dipping it into the fused flux. After either of these treatments the thickness of the aluminium coating is reduced by immersion of the article, when cool, in a solvent. Diffusion between the aluminium and the basis metal is achieved by heating at 1700°–2350°F. (925°–1288°C.). The flux and the aluminium are maintained at a temperature of 1280°–1400°F. (693°–760°C.) and 1250°–1325°F. (675°–718°C.), respectively.

Application of the treatment to blading of an alloy of the following composition is specifically claimed: chromium 13–17, iron 0·1–20, molybdenum 4–6, aluminium 1–6, titanium 1·5–3, manganese 1 max., silicon 1 max., boron 0·01–0·5, carbon 0·06–0·25, per cent., balance nickel.

GENERAL MOTORS CORPN., assignee of D. K. HANINK and E. R. PRICE. *Brit. Pat.* 809,922.
Similar to *Australian* „ 206,039.

See also *Brit. Pat.* 809,638, which covers the application of the process to components fabricated from alloys of cobalt base.

### Nickel-Aluminium Powder Components

Difficulties which have been experienced in production of nickel-aluminium powder composites are obviated by the finding that nickel amalgam can, in liquid mercury, be reacted with aluminium or aluminium amalgam, to form very fine mercury-free nickel-aluminium powders which contain 17–30 wt. per cent. of aluminium. Powders so produced are characterized by excellent sintering properties and the grain-size obtained is optimum for use in high-temperature components.

The reaction is effected at a temperature in the range 370°–750°C. (preferably 450°–750°C.). When cool, the floating nickel-aluminium powder is freed from mercury by decantation or filtration. Direct formation of nickel-aluminide powder with a NiAl phase occurs only if reaction between the starting materials is effected by heating within the range 500°–700°C., cooling, and re-heating within the same range. One heating results in formation of the $Ni_3Al$ phase and nickel powder.

The powders are stated to be suitable for compacting without further treatment, but metallic or non-metallic materials (e.g., $Al_2O_3$) may be added to improve or modify the properties of the resultant component, and binders (e.g., nickel amalgam, hydrolysable silicic-acid compounds, or alumina in colloidal solution) may be employed to facilitate shaping. The alloys are sintered, in a non-oxidizing atmosphere, at 1350°–1610°C.

The alloys made by this process are stated to be less brittle than cast alloys of similar composition, and to have higher hot strength and creep-resistance. This improvement is attributed to the presence of a skeleton of extremely fine NiAl particles: an alloy consisting of the NiAl skeleton with a binding phase of $Ni_3Al$ is stated to have given particularly good results.

SIEMENS PLANIAWERKE A.G. FÜR KOHLEFABRIKATE. *Brit. Pat.* 808,781.
Similar to *Canad.* „ 569,404.

### Sintered Nickel-containing Heat- and Corrosion-Resistant Alloys

Corrosion-resisting alloys having a density of 5·4–7·1 g./cc., high oxidation-resistance at temperatures in the region of 1800°F. (980°C.) and above, and good transverse rupture strength and stress-to-rupture values at elevated temperatures, consist essentially of a carbide phase (at least 30 per cent.) sintered with an alloy binder (20–70 per cent.). The preferred composition of the binder alloy is aluminium 3–25, nickel 65–95, molybdenum 3–30, per cent. The carbide phase includes titanium carbide (preferably 34–75 per cent.) and at least 2 (preferably 4–5) per cent. of niobium carbide.

J. W. GRAHAM, assignor to KENNAMETAL, INC. *U.S. Pat.* 2,867,033.

### Production of Nickel-Alloy-base Cermet Hollow Turbine Blades

A porous skeleton conforming to the configuration of the hollow component is first obtained by forming and sintering a body of refractory material around a core of high-melting material. This skeleton is then infiltrated interstitially with a matrix-forming metal or alloy of lower melting point. After cooling, the core is removed, giving a heat-resisting hollow component of the desired form.

The refractory skeleton consists of the following metals or their carbides, silicides, nitrides or borides: chromium, molybdenum, tungsten, vanadium, niobium, tantalum, titanium or zirconium. In application of the process to production of hollow gas-turbine blades, nickel, cobalt, iron, or heat-resisting alloys based on one or more of these metals, are employed as matrix materials. Typical compositions are quoted. Such blades are stated to exhibit satisfactory heat-resistance at temperatures in the region of 1000°C.

SINTERCAST CORPN. OF AMERICA. *Brit. Pat.* 809,133.

**Attention is drawn to the fact that many of the names of materials mentioned in The Nickel Bulletin are *Registered Trade Marks*.**

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